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The Journal *of the* Society of Dyers and Colourists

Volume 75 Number 5

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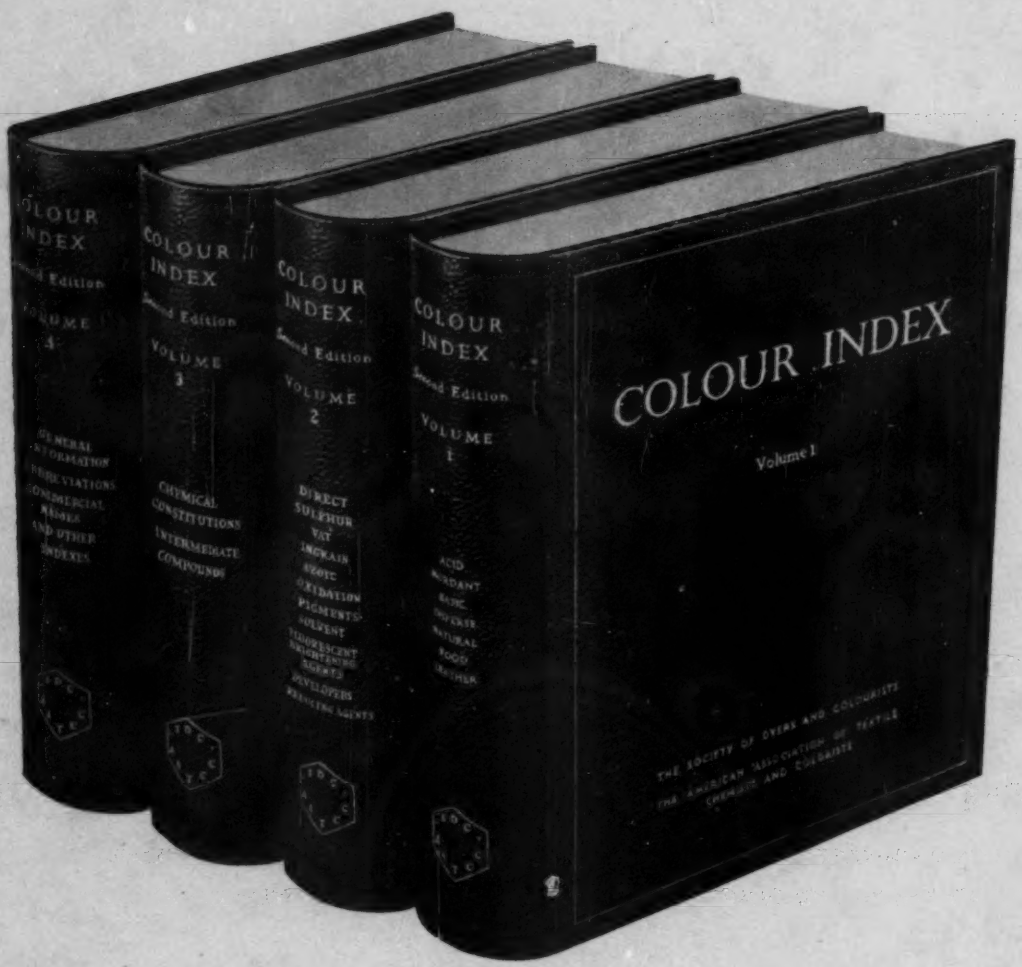

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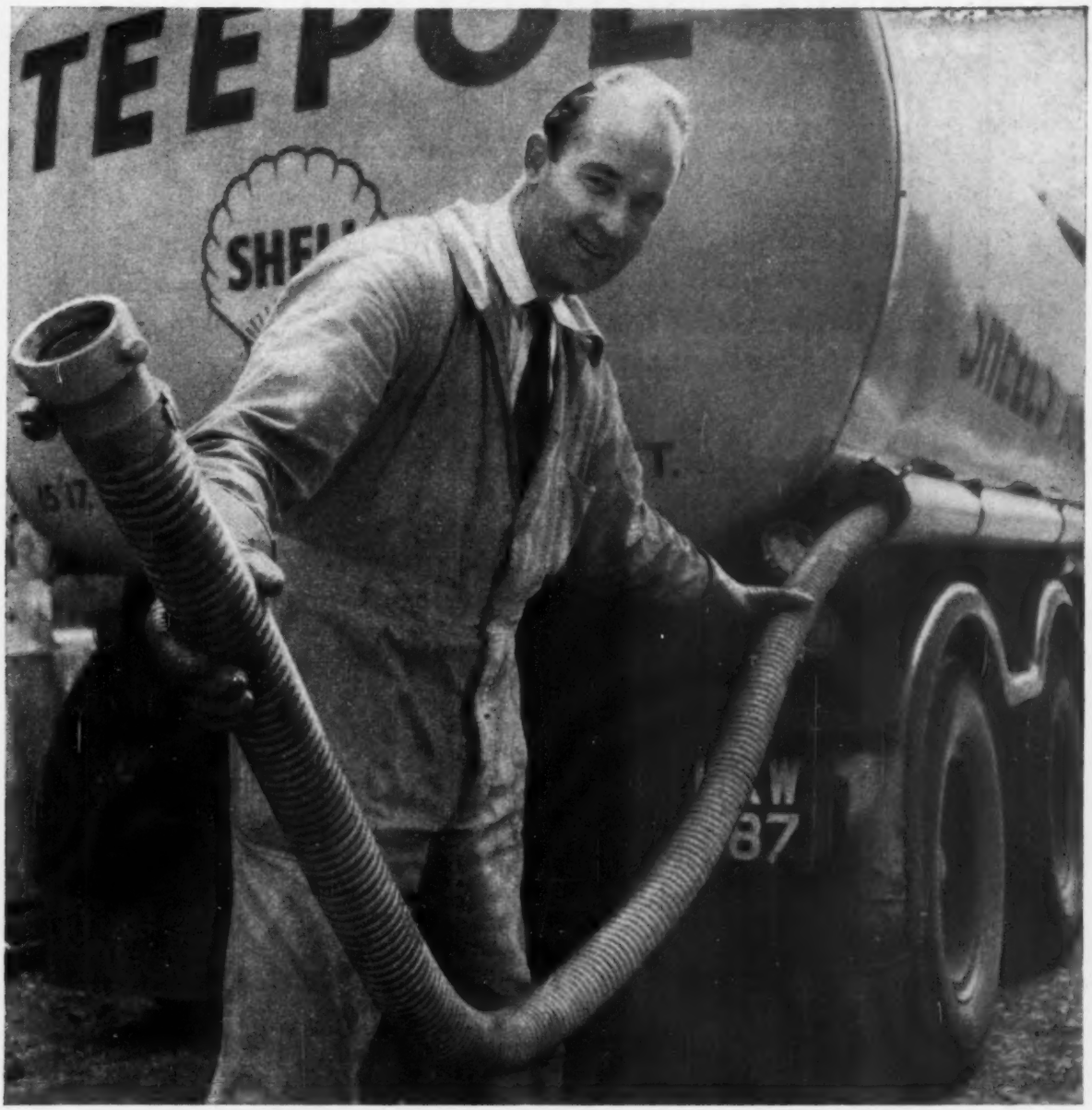

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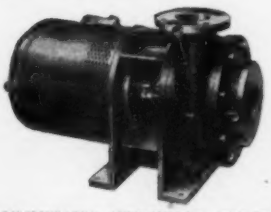

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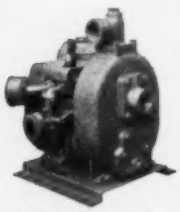

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NOTICE TO MEMBERS AND SUBSCRIBERS

Readers requiring *general information* regarding the Official Notices, List of Officers of the Society, etc, should consult pages 1–10 of the January 1959 and pages 509–516 of the July 1958 issues of the *Journal*, or write to *The General Secretary*, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire (*Telephone* Bradford 25138). *Editorial Communications* should be addressed to *The Editor*, at the same address

Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

LECTURES

The Mechanism of the Coupling Process	*W. Bradley*
The Life and Times of Peter Griess	*W. H. Cliffe*
The Diazo Reaction in the Coloration of Hydrophobic Man-made Fibres	*H. R. Hadfield and W. F. Liquorice*
The Dyeing of Polyester Fibres and Blends	*H. W. Partridge*
The Mechanisms of Diazotisation	*J. H. Ridd*
Relative Rates of Reduction of a Series of Azo Compounds	*G. P. Warwick*
The Finishing of Fabrics containing Wool blended with Terylene or Acrylic Fibres	*C. S. Whewell*

EXPLANATORY PAPER ON MODERN THEORY

Surface Activity, Solution, and Adsorption	*C. H. Giles*

FORTHCOMING MEETINGS OF THE SOCIETY

Friday, 29th May 1959

MANCHESTER SECTION. *The Degradation of Chemically Modified Celluloses by Alkali.* Dr. W. M. Corbett (British Rayon Research Association). The Textile Institute, **Manchester.** 7 p.m.

Friday, 23rd October 1959

LONDON SECTION. Seventh London Lecture. Waldorf Hotel, **London W.C.2.** 7 p.m.

Friday, 12th February 1960

LONDON SECTION. Annual Dinner and Dance. Waldorf Hotel, **London W.C.2.** 7 for 7.30 p.m.

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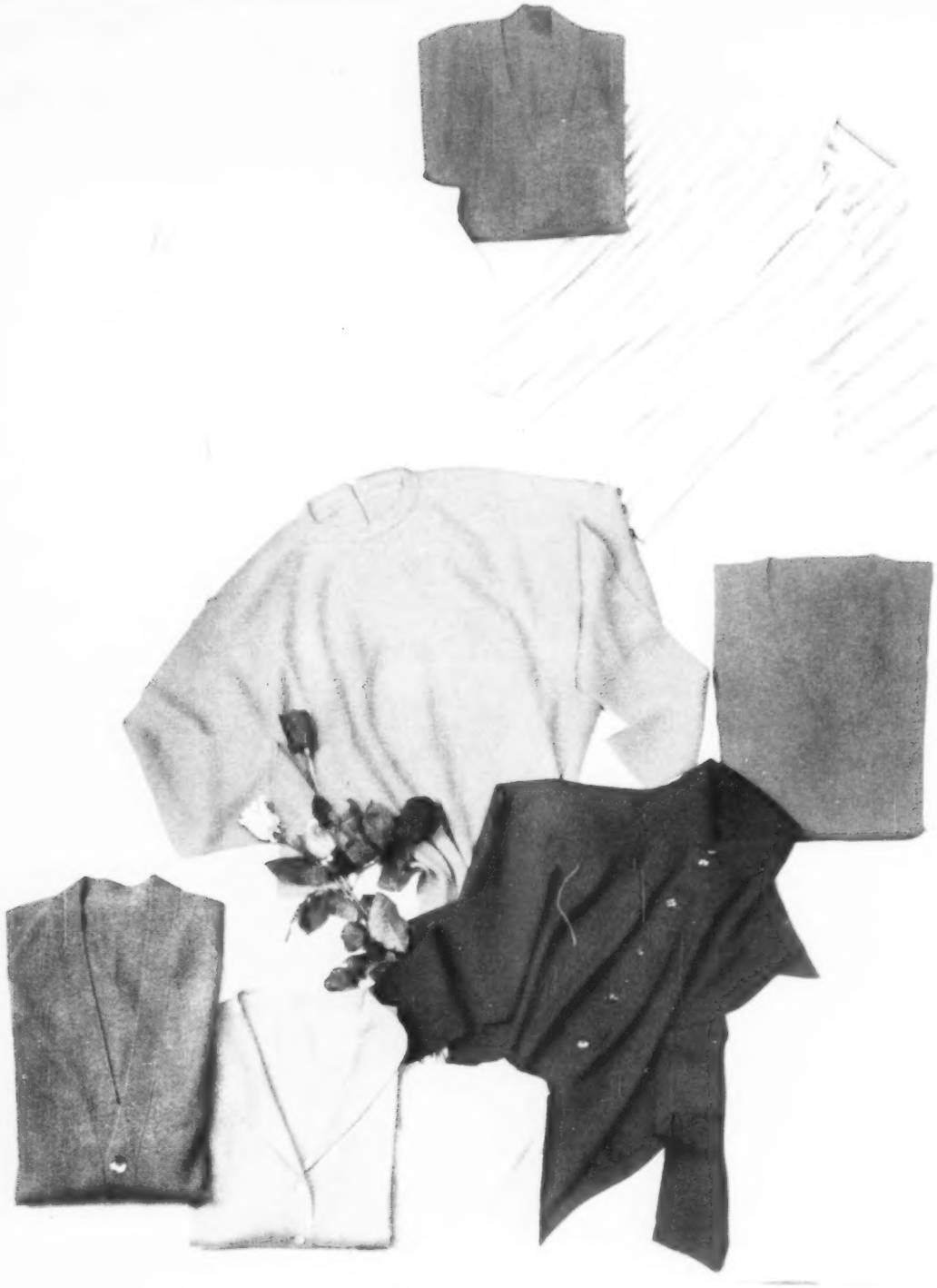

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Proceedings of the Society

Felisol

An International Informative Label for Colour Fastness

K. McLaren

Meeting of the Manchester Section held at the Textile Institute, Manchester, on 19th December 1958, Mr. J. W. Reidy in the chair

A historical account of the development of the International Association for the Felisol Fastness Label with special reference to the technical problems of issuing an informative label concerned with colour fastness.

In 1901 René Bohn, a Swiss chemist on the staff of BASF, attempted to make an indigoid dye containing an anthraquinone nucleus, as this had been found to confer valuable fastness properties on wool dyes. He therefore fused anthraquinonylglycine with caustic potash, and discovered, not another indigoid dye, but the first member of a much more important class, the anthraquinonoid vat dyes, which are unsurpassed for high fastness to washing. It was soon found that cotton dyed with indanthrone possessed high all-round fastness, so it was marketed as Indanthren Blue R, and is still one of the most important vat dyes. (C.I. Vat Blue 4).

The high all-round fastness properties were actually characteristic of many other anthraquinonoid vat dyes, so by 1910 about 100 brands were marketed, and it became feasible for goods to be sold bearing an unconditional guarantee in respect of colour fastness, as most would even withstand bleach at the concentration used in domestic washtubs.

Dyers, printers, and merchants were by no means unanimously in favour of these dyes, because they were more difficult to apply and increased the processing costs, and it could be argued that it was uneconomic to use a dye which lasted longer than the textile to which it was applied. To increase the demand for such dyes, the I.G. introduced what was undoubtedly the first informative label for colour fastness, the Indanthren label, which could be attached to goods dyed with Indanthren dyes, their leuco esters, and certain azoic combinations of similar fastness, provided that certain specified conditions were obeyed; the firm even opened shops, called *Indanthren Häuser*, which sold only cloth dyed or printed with Indanthren dyes. In 1952 the four firms which emerged from the old I.G.— BASF, Bayer, Cassella, and Hoechst— formed the Indanthren Trademark Association to continue the publicity which had been so effective: the consumer had found that goods bearing an Indanthren label did not fade and that the colour did not run in the wash, and discriminating shoppers were quite willing to pay a premium for this quality.

Dyers and printers in Continental countries had realised the advantages of the Indanthren label, but naturally did not want to have their dye purchases restricted to only one country (Germany), so they urged both the Fédération Internationale des Imprimeurs sur Tissus (F.I.I.T.) and the Association Internationale de la Teinture Textile (A.I.T.T.) to sponsor an international label.

After discussion, the A.I.T.T. came to the following conclusion: "It would be best if there were no fastness label, but since such a label already exists and impedes dye users in the purchase of their dyes, it is desirable to have an international label for cellulose and regenerated cellulose fibres, which guarantees at least the same type of fastness as the Indanthren label"[1]. At its meeting in Brussels on 10th July 1953, to which representatives of the dyemakers had been invited, two subcommittees were formed to carry out this task, one to consider the legal and financial aspects, and the other the technical requirements.

The development period was naturally full of difficulties, but these were overcome thanks mainly to the Swiss dyemakers, who persevered when negotiations within the A.I.T.T. had almost broken down, and in March 1956 the International Association for the Felisol Fastness Label* was founded with headquarters in Zürich. This body

* The trademark is spelt with capital letters— FELISOL.

is completely independent of the A.I.T.T., and its name is derived from the original French name, *F*édération du *L*abel *I*nternationale de *Sol*idité. Membership is open both to dye manufacturers and to dye users, who have equal powers in the running of the Association.

TECHNICAL CONSIDERATIONS

The Felisol label is initially restricted to natural and regenerated cellulosic fibres, for it is only for cellulose that there is a sufficient number of dyes of high all-round fastness which will enable most hues to be obtained, and which are made by most of the dye manufacturers selling in Western Europe.

For these fibres, the class of dyes needed for producing the fastest colours is the vat class, and the term "vat-dyed" is used occasionally as an informative label in this country; it is more widely known in the U.S.A., where an organisation, the Vat Dye Institute, has been formed "to spread the knowledge about Vat Dyes—the fastest group of dyes known to science". However, the fact that vat dyes have been used is not in itself an indication of high colour fastness, for several reasons which are worth considering in detail.

In the first place, one of the oldest dyes—Indigo—is a vat dye, but on cotton its fastness to light is only 3 even at standard depth; its fastness to washing is also only moderate, as evidenced by the loss in depth of sailors' collars on repeated washing; the newer indigoid dyes, although of better fastness, still leave much to be desired.

This objection could be overcome by referring only to anthraquinonoid vat dyes, which could be publicised as "AQ-Vats", but whilst the fastness properties of this class are much higher, there are a significant number unsuitable for certain end-uses—

(1) Those which sensitise the photodegradation of cellulose, called "tenderers", are unsuitable for curtains

(2) Those whose washing fastness leaves something to be desired, e.g.—

C.I. Vat Red 23
C.I. Vat Violet 14
C.I. Vat Blue 12

are unsuitable for goods which will receive heavy and repeated launderings

(3) Those whose fastness to weathering is insufficient for them to be used for awnings.

In addition, many dyes are unsuitable for curtains if dyed in pale colours, as light fastness almost always decreases with decreasing concentration and may reach dangerously low levels. Another factor which prevents the term "AQ-Vat" from being satisfactory is that the range is noticeably deficient in reds; these can, however, be obtained with the same fastness properties from the azoic combinations. A "vat-dyed" label would similarly exclude dyes such as Alcian Blue 8GX (ICI) (C.I. Ingrain Blue 1), which are indispensable for producing the popular turquoise blues.

The Technical Subcommittee, therefore, first decided that cellulosic textiles would have to be divided into three main groups according to end-use, as the number of dyes suitable for all purposes is too small; the three groups are—

Group 1—Washable materials, where washing fastness is the main requirement—

(*a*) Articles receiving severe and often repeated washing in use, e.g. shirts, table and bed linen, overalls

(*b*) Articles receiving only mild and occasional washes, i.e. dress fabrics.

Group 2—Indoor materials where light fastness is the main requirement—furnishing fabrics, curtains, and upholstery fabrics.

Group 3—Outdoor materials where weathering fastness is the main requirement—awnings, tent cloths, etc.

At its early meetings the subcommittee drew up an impressive provisional list of minimum fastness gradings for each of these groups, not only to light and washing, but also to rubbing, perspiration, hot pressing, soda boiling, hypochlorite, and peroxide bleaching. During the following three years the individual dyes of the interested firms were classified according to their suitability for each of the three end-uses, and at the same time the minimum gradings were constantly being modified in the light of experience. Finally, it was decided to reduce the *official* fastness requirements for any new dyes to light, washing, and weathering. It should be noted that there is a minimum light fastness for washable materials, and also a minimum washing fastness for furnishings.

Light fastness is determined by the ISO method by exposure to daylight, the use of fading lamps being prohibited. Washing fastness is determined by Test C of the E.C.E., which is now identical with I.S.O. Mechanical B test.

The fastness requirements which govern the selection of dyes are published in the Technical Regulations, which are given to all members. As examples, the following may be quoted—

Indoor Materials—Light fastness at least 6 at 1/1 Standard Depth in the case of vats, etc.; 6–7 in the case of azoics.

Washable Materials (*Group 1a*)—Washing fastness at 1/1 Standard Depth: effect on the specimen 4, staining 4.

The question of suitability for outdoor materials was particularly difficult, because there is no suitable recognised test for fastness to weathering in any of the world's fastness testing systems. Deciding on suitability for this class of goods for Felisol undoubtedly gave considerable impetus to the development of a suitable test, e.g. the papers of Brunnschweiler [2] and of the present author [3], who in fact used the Felisol classification, based on the cumulative experience of all the participating dyemakers, as proof of the reliability of the method of exposure against the light-fastness standards exposed simultaneously, but under glass.

The restriction of the official requirements to light, washing, and weathering does not mean that

any dye which reaches these minimum requirements is accepted into the Felisol system, because each dye is individually considered by the Technical Committee, and if it possesses defects which are revealed only by other, non-specified tests, it may be rejected for one or more of the three classes of suitability. A hypothetical and extreme case would be a dye which was rated 8 to light and 5 to washing but which was completely removed from the fibre under normal conditions of dry cleaning. Actual examples of this principle in action are naturally less spectacular, but one or two are worthy of mention—

(1) Caledon (Printing) (Brilliant) Blue 3G (C.I. Vat Blue 12) is permitted for printed washable materials but not for dyed washable materials, as it has a tendency to stain under severe washing conditions; these conditions rarely occur with printed fabrics

(2) Those sulphur dyes which meet the official requirements have not been admitted with the exception of Carbindone Black B (C.I. Sulphur Black 11); this is because of their well known limitations, e.g. very poor fastness to dilute sodium hypochlorite, tendering tendencies, poor stability on storage.

Conversely, one or two dyes which do not meet the official requirements have been admitted because their exclusion would unduly restrict the dyer and the printer in meeting the legitimate wishes of textile designers. For example, bright pinks and scarlets could originally be obtained only by using indigoid vat dyes (whose defects were mentioned earlier), so they have been admitted, but their use has been restricted. Durindone Pink FF (C.I. Vat Red 1) can be used only for printed furnishing fabrics, as the fading of one colour in a multicoloured print is much less noticeable than that of a self colour. The use of Durindone Printing Scarlet Y (C.I. Vat Red 45) is even more restricted: whilst C.I. Vat Red 1 can be used for dyed and printed washable materials and printed furnishings, C.I. Vat Red 45 can be used only for printed dress goods, as these are not, as a rule, subjected to severe washing conditions.

The minimum light fastness demanded for azoic combinations is significantly higher than that for vat dyes: for washable materials, the minimum light fastness is actually higher than that for vat-dyed furnishing fabrics. This is partly because, with the thousands of possible combinations, a higher level (which is obviously desirable) is practicable, and also because the fading rates of azoic combinations are generally much more sensitive to humidity, so that additional safeguards are essential.

It is well known that the light fastness of most dyes is lower in pale colours, and therefore it was essential that the user should be prevented from dyeing and printing pale colours whose light fastness fell below the minimum level. This was ensured by establishing where necessary a minimum depth, whose light fastness equalled the minimum level, and every user is forbidden to use that particular dye in any weaker depth. There is a minimum depth for every azoic combination.

In theory, of course, there will be a different minimum depth for each style, i.e. some dyes will need three, but the determination of them would have taken so long that it was felt unnecessary at first. So the minimum depth selected was for that group where light fastness is most important, viz. furnishing fabrics. It was also felt that the fact that the dyer or the printer could not use a slightly weaker colour for washable materials would not be particularly frustrating.

For outdoor materials, minimum depths have been established only for azoic combinations: all other dyes which are suitable must be used "in full shades".

When the Technical Committee had decided for which classes the dyes made by the manufacturing members were suitable, what the minimum depth (if any) should be, and what other restrictions concerning use were considered necessary, the dyemakers then began the preparation of the scripts and the cloth for the pattern cards. These were all made by one firm of pattern-card makers in Switzerland and were of identical format. Each contained not only "bread and butter" dyes common to several makers but also specialities, so that a progressive user needs a complete set. The larger dye firms issued two cards, one for dyers and one for printers, whilst the smaller issued only one in two sections. The total number made was 30,000.

The cards are very simple to use, obvious symbols being used to indicate suitability for each of the three main classes of end-use. Instructions are printed in five languages— English, French, German, Italian, and Dutch— and each contains the most important items from the Technical Regulations.

The cards are in loose-leaf format, enabling supplements to be issued when new products are added to any manufacturer's range which have been accepted by the Association. Two hundred new applications have been submitted since the original list closed in April 1956.

THE FELISOL ORGANISATION

The International Association for the Felisol Fastness Label is incorporated as an association under the Swiss Civil Code, with its registered headquarters in Zürich. Membership of the Association is open to dye manufacturers who produce and sell dyes on the approved list and to a National Organisation (N.O.) of dyers and printers in each country. A National Organisation is formed if a minimum number of six users are willing to join; if this number cannot be reached, then individual users can join and use the label, but they have no voting power. The membership of the Association is* as follows—

Dye manufacturers	16
National Organisations	6
Holland	57 members
Italy	46 members
Switzerland	33 members
Austria	17 members
Belgium	17 members
Spain	9 members

* April 1959.

together with one United Kingdom and eleven French associate members.

Dyers and printers have to sign a declaration that they will comply with all the regulations of the Association, one of which stipulates that a fine of up to £500 can be imposed for any breach. Any differences are settled in equity according to the procedure of arbitration of the International Chamber of Commerce.

The governing body of the Association is the General Meeting, which normally meets once a year; at these meetings the manufacturers' and National Organisation groups each have the same number of votes. General management of the Association is conducted by an Executive Committee of at least eight members (there are twelve at present), half of whom represent the makers and half the users; they are elected by the General Meeting for a two-year term. The General Meeting also appoints the Technical Committee, whose members are not limited in number nor in duration of appointment; voting in this Committee is also equally divided. The Association also has salaried secretarial staff and auditors.

The Association places the Felisol label, which exists in several forms, at the disposal of the National Organisations, which may also grant the right to use it to firms dealing in textiles, at stages subsequent to dyeing and printing, e.g. clothing manufacturers, providing that they accept the same regulations. The Association provides the National Organisations with labels, each of which bears a number, so that in the event of a complaint, the dyer or printer can be identified. The dyer or printer must retain for at least two years a cutting of each recipe used.

The Executive Committee designates independent testing laboratories, which may be asked by the National Organisation to carry out identification and fastness tests on complaints from the public, which can be made either to the dyer or printer, to the National Organisation, or to Felisol headquarters. The National Organisations also appoint inspectors, who have the power to demand cuttings from goods bearing the label from both wholesalers and retailers; the number of test samples to be taken is decided by the Secretary of the Association, and is proportional to the number of labels issued in the country concerned.

The Felisol label received its first publicity in the advertisements of some of the participating dye manufacturers in November 1957. 80,000 copies of an introductory brochure printed in five languages have been distributed, and in May 1958 an advertising campaign aimed at the general public was launched in Italy: thirty-six advertisements appeared in ten daily papers at the time of the general election, when readership figures reached their maximum. In September coloured advertisements appeared in the monthly magazines *Schweizer Illustrierte Zeitung* and *Illustré*, and a colour film has been produced for general showing in cinemas. In Holland, articles describing the Association have appeared in the technical press. An information bulletin in three languages is also regularly distributed, and so far (March 1959) seven issues have appeared. This now lists dyes recently accepted into the Felisol system which are not yet in the Felisol pattern cards.

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(*MS. received 8th January 1959*)

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Discussion

Mr. G. S. Marr: As a commission processer, I agree with the principle of the Felisol scheme on three counts—

(1) It appears to safeguard producer and consumer alike

(2) It is a logical sequence to all the international work on colour fastness

(3) It fits precisely into the context of European Free Trade.

Although I welcome the Felisol principle, I am not blind to the practical difficulties of the scheme nor to the objections that will be raised. I can well imagine those merchants, manufacturers, piece finishers, and retail distributors who have established exclusive marks of quality taking initial umbrage at the prospect of a universal warranty, but I think that this will pass and that the Felisol mark will be accepted, not as an alternative, but as an additional warranty which confirms the exclusive quality mark.

The real problem, however, lies in the horizontal system of production which is largely characteristic of the Lancashire textile trade, in the operational mechanics and responsibilities of passing the Felisol identity from one section to another, and not least in the financial obligations involved. It is no simple matter. There are many ramifications, particularly so in the case of yarn and loose fibre. I do not suppose that the problem is insuperable, but it does need a lot of detailed thinking. Can Mr. McLaren make any helpful comment to this end?

Mr. McLaren: The operational mechanics in any one country cannot be worked out by Felisol headquarters, and this task is the responsibility of the National Organisation as soon as it is formed, because only the members of this body understand the detailed ramifications of their own country's trade. The horizontal system in Lancashire is not, however, unique: there is a lot of commission dyeing and printing in Switzerland, and doubtless these problems are being studied by the Swiss National Organisation.

Dr. C. M. Whittaker: Are pigments used in mass coloration on the list of approved dyes? Carbon Black and Monastral Fast Blue, for example, would equal anything on the approved list.

Mr. McLaren: At present, mass-pigmented viscose rayon is excluded from the Felisol scheme, but the question of its admission is being considered by the Association.

Dr. Whittaker: In assessing the fastness to light of the approved dyes, has the deteriorating effect of some fluorescent brightening agents on them been considered?

Mr. McLaren: Yes. The use of fluorescent brightening agents is forbidden except on bleached, undyed yarns that are to be used in colour-woven fabrics and in white-resist and white-discharge printing styles.

Mr. F. Cusack: Is the use of the Felisol mark confined to goods processed with dyes purchased from dye manufacturers who are members of the Association? If so, is the mark in fact a certification trade-mark duly registered under Board of Trade rules in the United Kingdom?

Mr. McLaren: Only dyes made by manufacturer-members of the Association can be used if the goods are to receive a Felisol label. The Felisol name and the design of the label were originally registered in this country by Ciba Ltd. on behalf of the Association, but I do not know under which category it comes at the moment or will in the future.

Mr. A. H. R. Dowdeswell: To what extent has the Felisol scheme found favour in England up to the present time?

Mr. McLaren: Imperial Chemical Industries Ltd. and L. B. Holliday & Co. Ltd. are manufacturer-members, and several dye users have written to Felisol headquarters for information. No English firm has yet applied for membership, but a Northern Ireland firm has.

Mr. B. Kramrisch: Has any approach been made to such organisations as the Good Housekeeping Institute?

Mr. McLaren: No. Felisol is an informative label in its own right, and no attempt will be made to get any other quality control organisation to adopt it.

Mr. A. F. B. Nall: How is it proposed to co-ordinate Felisol criteria with those which might be considered for inclusion in national (or international) performance standards?

Mr. McLaren: Felisol criteria are unique (apart from Indanthren) in that, not only are levels of fastness fixed, but also only dyes on the permitted list can be used. This would probably make co-ordination with national performance standards impossible.

Mr. Kramrisch: Where the Felisol label is to be applied to an all-cotton garment including linings and sewing threads, what will be the position regarding buttons and other trimmings?

Mr. McLaren: The International Association specifies that the label can be attached to garments only if all the coloured textile components bear the label. No regulations governing non-textile components have been laid down by the International Association, but could be by the National Organisations.

Mr. Nall: How are the members of the Executive Committee appointed and how is a National Organisation formed?

Mr. McLaren: The members of the Executive Committee are appointed by the Annual General Meeting for a two-year period, each member being eligible for re-election. A National Organisation is formed by discussions between representatives of the user-members in the country concerned and the Secretary of the International Association. The National Organisation appoints its own Management Committee, which runs the National Organisation and appoints its representatives on the two committees of the International Association.

Dr. C. J. W. Hooper: Are the Americans co-operating in the Felisol Association?

Mr. McLaren: Yes. E. I. du Pont de Nemours & Co. and General Dyestuff Co. are members; no interest has yet been shown by dye-users in the U.S.A.

Dr. Hooper: Do the colorants used for the Felisol labels conform to the Felisol standards?

Mr. McLaren: The cloth labels are made from yarns bearing the label. The main labels are printed cards, and three printers submitted specimens, whose light fastness varied from 1 to 6. The Association chose the card of highest fastness, which, incidentally, was printed in a much brighter yellow.

Dr. Hooper: Are C.I. numbers to be used for the list of permitted dyes?

Mr. McLaren: The new *Colour Index* appeared after the list had been drawn up, but there does not appear to be any need to include C.I. numbers today.

Mr. J. A. Clarke: Does the minimum depth shown in the pattern cards mean that that dye must be used in equivalent concentration in combination dyeings, and has the effect of one dye on the fastness properties of another in combination dyeings been taken into account?

Mr. McLaren: Minimum depths must be followed in combination dyeings. Catalytic fading has been avoided by prohibiting the use of the worst offender, C.I. Vat Yellow 2, except up to a certain amount with specified blue dyes for washable materials only.

Dr. Whittaker: From my experience of textile designers, I would suggest that they will take no notice whatever of the specified minimum depths.

Mr. P. R. Dawson: Is the Indanthren label in competition with the Felisol label, and if so, what is the attitude of the Germans towards it? Is there any restriction on their participation in the scheme?

Mr. McLaren: The Germans have co-operated wholeheartedly with the other countries in the creation of the Felisol label and they were founder members. The Indanthren label is still extant, however, the Germans benefiting from both labels. They have not, as yet, followed the other manufacturers and included the Felisol mark in their publicity.

Mr. W. W. Burgess: Would a printer who was a member of the Felisol Association be permitted to incorporate the word Felisol in a printed selvedge legend?

Mr. McLaren: Yes, providing that the selvedge marking also included the printer's number or other means of identification.

Dr. HOOPER: Is a fine of £500 imposed by a foreign organisation on a British firm likely to be upheld by the English and Scottish courts?

Mr. MCLAREN: I have not been concerned with the legal side of Felisol, but according to the bye-laws of the Association, differences of this kind "shall be settled in equity, to the exclusion of ordinary courts, according to the procedure of arbitration of the International Chamber of Commerce before one or several arbitrators appointed according to the rules of the International Chamber of Commerce".

Methods of Assessing the Dyeing Properties of Wool Dyes*

B. KRAMRISCH

Meeting of the Huddersfield Section, held at Silvio's Café, Huddersfield, on 21st October 1958, Mr. H. Boothroyd in the chair

The importance of the dyeing behaviour of wool dyes necessitates the availability of suitable methods for assessing such properties as migration, rate of strike, etc., including the Society's own recommendations [1].

MIGRATION TEST

The S.D.C. migration test for wool dyes [1] has now been applied to three acid yellows, three acid reds, and three Kiton Fast Blues (C.I. Acid Blues 47, 45, and 50), each group consisting of a mono-, di-, and tri-sulphonate. The results show that migration is favoured by a low degree of sulphonation, in agreement with earlier results [2] on rate of dyeing.

Although the migration test was originally worked out for acid dyes [1], it has now been applied to 1:1 metal-complex and chrome dyes, and some of the results are shown in Tables I and II. They bear out the behaviour of these dyes in practice.

A test of this type is, however, of no value in the case of the 1:2 metal complexes (Cibalans), as virtually no migration takes place with this class of dye. A large number of dyes of this category are very level dyeing, and many can actually be "salted" at the boil. They owe this behaviour primarily to an even strike, and it was felt, therefore, that a test to incorporate this property was of importance.

COMBINED STRIKE–MIGRATION TEST

A combined strike–migration test for 1:2 metal-complex dyes was worked out in our laboratories concurrently with a similar type of test, although not quite identical in detail, to that developed by Lister [3]. Details of our test are as follows—

The dyebath is set with the requisite amounts of dye and assistants calculated for 10 units of material, and is brought to the boil. The first piece of material, weighing 4 units, is entered; further pieces, each weighing one unit, are then entered at intervals of 2, 4, 8, 16, 32, and 64 min. respectively after the introduction of the first piece; and boiling is continued for a further 56 min. On completion of the dyeing, the patterns are ironed, trimmed, and mounted side by side in the order of addition to the dyebath, and the dyeing behaviour of the dye under examination is evaluated by visual examination of the series of patterns.

A gradual "tailing-off" of the patterns indicates a dye with good strike–migration properties. The test is also useful for assessing the behaviour of dyes in mixtures. For instance, if this test is applied to combinations of Cibalan Yellow FGL and three different "blue" dyes—

Cibalan Grey BL (C.I. Acid Black 60)
Cibalan Blue 3GL (C.I. Acid Blue 171)
Cibalan Blue BL (C.I. Acid Blue 168)

it clearly shows marked differences between the three combinations. Cibalan Yellow FGL and Cibalan Grey BL are a very good pair as regards levelling and compatibility, being superior to Cibalan Yellow FGL and Cibalan Blue 3GL, which

TABLE I

Migration Properties of Neolan Dyes on Wool and Acrilan

Dyeing		*Colour Index* Designation	Migration Figure [1] Wool	Acrilan
2·3%	Neolan Pink BA	C.I. Acid Red 186	3	–
1·7%	Neolan Red BRE	C.I. Acid Red 212	4	–
1·8%	Neolan Light Brown C	–	2	3
1·3%	Neolan Bordeaux BE	–	3	3
2·6%	Neolan Dark Blue F2R	–	4–5	4

TABLE II

Migration Properties of Chrome Dyes on Wool

Dyeing		*Colour Index* Designation	Migration Figure [1] Unchromed	Chromed
0·8%	Chrome Fast Yellow G	C.I. Mordant Yellow 16	1	1
1·0%	Chrome Fast Flavine A	C.I. Mordant Yellow 5	4–5	4–5
3·0%	Chrome Fast Cyanine 2BSS	C.I. Mordant Blue 5	2	2
2·75%	Chrome Fast Blue ACB	C.I. Mordant Blue 9	3	3

* Summary of lecture

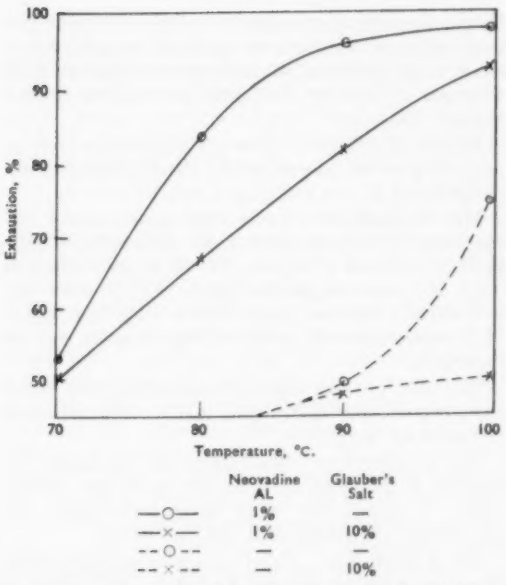

FIG. 1—Temperature-range Curves for 2% Cibacron Scarlet 2G on Wool (+ 2% acetic acid for 1 hr.)

in their turn are superior to the same yellow with Cibalan Blue BL. These findings bear out the known properties of these dyes.

The same test has been found useful in evaluating dyeing assistants with 1:2 metal-complex dyes and also in evaluating dyes from the Alizarine, Benzyl, and Chrome ranges. In the case of the latter, dyeing is carried out as specified above, and the dyed materials are then after-chromed with the usual reagents at the boil for 30 min.

TEMPERATURE-RANGE AND RATE-OF-DYEING CURVES

These curves often give useful information.

An example of the use of temperature-range curves is given in Fig. 1, which illustrates the behaviour of a reactive dye, Cibacron Scarlet 2G, on wool under various conditions. It indicates how the uptake of dye is facilitated by the addition of Neovadine AL to the acetic acid dyebath and also the levelling action of Glauber's salt.

Rate-of-dyeing curves illustrate how different dyeing behaviour can be, even with dyes of similar chemical constitution. They are also of value for comparing the behaviour of a dye under various dyebath conditions, e.g. with different assistants in the bath. Thus Fig. 2 shows that, taking into consideration economic bath exhaustion, preference could be given to Glauber's salt alone over acetic acid or ammonium acetate in controlling the uptake of Benzyl Cyanine 6B (C.I. Acid Blue 83), other factors being equal.

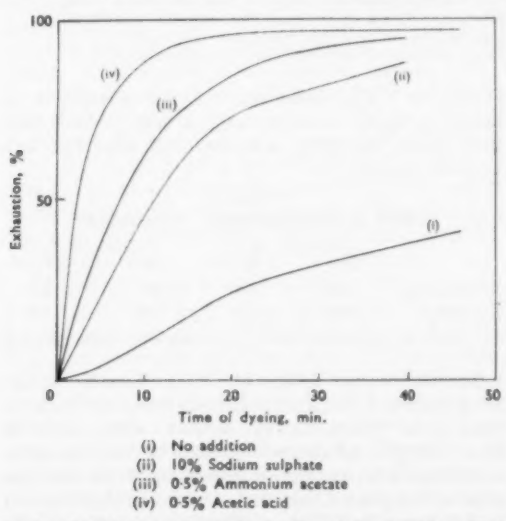

FIG. 2—Rate-of-dyeing Curves for 1% Benzyl Cyanine 6B on Wool with Various Assistants (hosiery yarn in 50:1 liquor at the boil)

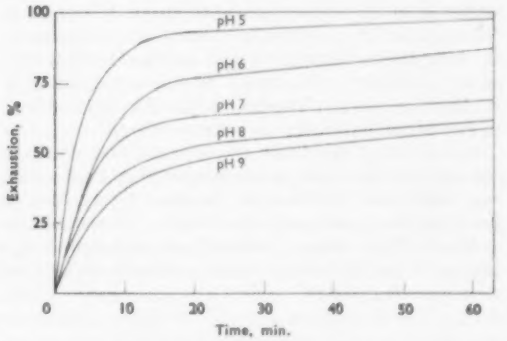

FIG. 3—Rate-of-dyeing Curves for Cibalan Grey BL on Nylon at Different pH Values

Fig. 3 illustrates the effect of pH variation in controlling the rate of dyeing of a 1:2 metal-complex dye, Cibalan Grey BL (C.I. Acid Black 60), on nylon.

* * *

I acknowledge my indebtedness to CIBA Clayton Ltd. for permission to give this lecture and to several colleagues, including Mr. D. G. Evans, Mr. T. Green, and Mr. G. P. Davies, for their assistance.

CIBA CLAYTON LTD.
MANCHESTER 11

(*MS. received 27th October 1958*)

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Discussion

Mr. M. NUTTON: Has the strike–migration test been tried out on piece material which has been damaged by over-carbonisation?

Mr. KRAMRISCH: We have not carried out such tests.

Mr. R. L. ROBINSON: What are the main drawbacks and advantages with the Cibacron dyes on wool?

Mr. KRAMRISCH: The main use of Cibacrons on wool is likely to be for bright reds, scarlets, and oranges owing to the brightness and high standard

of light fastness obtainable. Some staining of white cotton is liable to occur when the dyeings are subjected to washing and perspiration under alkaline conditions, but this can be minimised by including an alkaline treatment at the end of the dyeing process.

Mr. G. ENGLAND: Does temperature control come into the strike–migration test?

Mr. KRAMRISCH: The dyeings are carried out at the boil under comparable conditions.

Mr. ROBINSON: Are further developments likely in the field of reactive dyes for wool?

Mr. KRAMRISCH: Recent developments are exemplified by the introduction of the Cibacrolan dyes, which, although not strictly reactive, have been developed specifically for wool. Bright turquoises and greens are obtainable with the addition of an organic acid (acetic or formic) and Neovadine AN, and these colours are comparable in brightness with dyeings produced with triphenylmethane acid dyes. The fastness to light is, however, much superior with the Cibacrolans, being of the order of 5 in average depth.

Dr. F. MANCHESTER: How were the dyebath exhaustions assessed in the graph (Fig. 1) showing the behaviour of Cibacron Scarlet 2G applied to wool by the acetic acid–Neovadine AL method?

Mr. G. P. DAVIES: Initially the exhausted dye liquors were estimated colorimetrically using the Hilger Spekker photoelectric colorimeter, but the final results were obtained by visual comparison of the dyeings produced at various temperatures with a range of dyeings prepared at various known depths.

Mr. H. E. WOOD: Can the migration test be used to give an assessment of the milling fastness of dyes?

Mr. KRAMRISCH: In a very general way the migration test does give some indication of the milling fastness of dyes. For example, where a wool dye has a migration figure of 5, it often has low milling fastness; and with a migration figure of 1, comparatively good milling fastness can be expected.

Mr. WOOD: Can some indication be given of the function of the Neovadine AL in the Cibacron dyebath for wool?

Mr. KRAMRISCH: Neovadine AL promotes the uptake of Cibacron dyes by the wool, but at this stage I would not like to put forward any theoretical explanation of this behaviour.

Mr. KRAMRISCH*: Casty[4] has now suggested that Neovadine AN (similar to Neovadine AL), which is cation-active in an acid bath, forms a complex with the dye covering the fibre surface, from which the dye slowly and uniformly penetrates to the interior.

* Communicated.

COMMUNICATIONS

Cellulose and Formaldehyde

J. T. MARSH

The reaction between cellulose and formaldehyde has been known for over fifty years and, whilst its technical application is full of pitfalls, there are many aspects of great theoretical interest in connection with the chemistry of cellulose and the mechanism of certain finishing effects. One type of reaction with cellulose in the swollen state gives a product with "wet but not dry" recovery from creasing, whereas another type of reaction with the collapsed fibre gives a damaged product with high crease-recovery and low water-imbibition. The results accord generally with the proportions of regions of poor lateral order in cotton and rayon, and bear some relation to similar effects produced by resinous condensation products. The effect of the process on the breaking loads of cotton and rayon presents some anomalies, which are discussed together with the controversial matter of water-imbibition and covalent cross-linkages.

It is now more than fifty years since Eschalier[1] patented his process for the strengthening of regenerated cellulose by treatment with formaldehyde; it was known as *sthenosage*, from the Greek σθενος, "strength". The usual method was to impregnate the cellulose with an acidified solution of formaldehyde, dry, and then heat at a moderately elevated temperature. Most subsequent investigations have followed this plan, but it is quite clear that the process is very difficult to control, because the reaction with the cellulose is in competition with the evaporation of the formaldehyde. Although this process was very well known, it never attained commercial success.

However, the fact that the regenerated cellulose could be strengthened in this way was substantiated by Cross and Bevan[2], who found that the amount of combined formaldehyde in the final product lay between 0·35 and 0·8%; they also provided some data on the mechanical properties (Table I). The reduction in the extension at break is quite evident, and it was stated that the dyeing capacity was reduced also but was very irregular.

TABLE I

Effect of Formaldehyde Treatment [2]

	Strength (g.)*		Extension (%)*	
	Dry	Wet	Dry	Wet
Untreated	1·25	0·37	9·0	12·2
Treated	1·6	1·1	7·6	7·8

* It should be remembered that these values refer to the rayons of 1908.

In 1919 it was found by F. L. Barrett, in the laboratories of the Tootal Broadhurst Lee Co. Ltd., that the treatment of cotton with acidified formaldehyde solutions, followed by heating, gave a product with very good crease-recovery, but this was accompanied by such severe embrittlement and reduced tensile strength as to be commercially valueless[3].

In the autumn of 1925 it was shown by the writer that when cotton was treated with acidified formaldehyde in non-aqueous solvents, but without any subsequent heating, a reaction took place which gave a product of good immunity to dyes, but once again the cotton was so severely damaged that it was considered to be unmerchantable.

Repetition of Eschalier's process by Bruckhaus [4] gave a product which dyed more deeply than the original material, in contrast to previous results, but Bruckhaus heated only at 60°C.; this is an important point, as will appear later.

The publication of Meunier and Guyot [5] came at a time when the linear structure of cellulose was being formulated, and from their measurements of water-retention after centrifuging the wet, treated samples they concluded that the reduction in swelling was due to blocking of the hydroxyl groups of the glucose residues; but because this reduction was so great, and was accompanied by an increase in strength, they suggested that there must be a cross-linkage between adjacent molecular chains or between adjacent crystallites. This conclusion was based almost entirely on the evidence of reduced water-retention.

Another examination of Eschalier's process, by Wood [6], showed quite clearly that there are two distinct stages in the reaction. In the first stage the rayon fibres are insoluble in cuprammonium hydroxide but absorb large amounts of copper and dye deeply; in the second stage, the fibres are also insoluble in cuprammonium but they do not swell, nor absorb the reagent, nor dye as deeply as the untreated rayon. Hence the water content and the rate of reaction determine the effect produced; this, as pointed out by Wood, explains the deeper dyeing effects of Bruckhaus, who cannot have employed conditions of time and temperature to give a low moisture content.

The reaction between cellulose and formaldehyde in presence of strong sulphuric acid also was examined in the early 1920s. Samec and Ferjancic [7] used 60–75% H_2SO_4 containing 5–10% formaldehyde, whilst Barrett and Foulds [8] used smaller amounts of formaldehyde to control the action of strong sulphuric acid on cotton. This type of reaction was also examined by Wood [6], who found it possible to combine up to 7% of formaldehyde with cotton by treatment with about 73% H_2SO_4 and 4·8% formaldehyde; the product was found to swell greatly in water, to an extent comparable with methylcellulose. This type of cellulose methylene ether exhibits strong crease-recovery in the wet state but not when dry.

Following upon the invention of the crease-resisting process by Foulds, Marsh, and Wood [9] in the laboratories of the Tootal Broadhust Lee Co. Ltd., considerable activity was displayed elsewhere in investigating the possibility of obtaining similar effects by the direct action of formaldehyde, both on cotton and on rayon [10]. Several of these processes have been discussed by Marsh [11]. In general, the unpublished conclusion of Barrett was confirmed, viz. that, although crease-resisting effects could be obtained on cotton, the amount of degradation was so great that the goods were of no commercial value; a loss in tensile strength of 66% was reported. The crease-resisting effect on rayon also was accompanied by severe damage. It is clear from these specifications, too, that attempts were made to utilise some of the work in anti-shrink processess, but even here there was excessive damage on cotton, although some success was obtained with rayon. Advice was given that the amount of combined formaldehyde should lie between 0·1 and 0·4% for cotton and between 0·1 and 1·5% for rayon.

Many subsequent investigators appear to be unaware of the intensive work done between 1932 and 1939, but it is quite clear from these patent documents, which form part of the literature on the subject, that the reactivity of rayon is greater than that of cotton, and that the degradation of cotton is greater than that of rayon and much greater than might be expected.

The first systematic quantitative work to be published was that of Saegusa [12] (1941); the writer received an intercepted copy of his paper, and was fortunate in being able to arrange for its translation. Saegusa worked with rayon, which was impregnated with formaldehyde solution at pH 1·7, dried, and heated at 70°C. for 5 hr. His conclusions were that crease-recovery required at least 2% combined formaldehyde, but there was no further improvement beyond about 5% combined formaldehyde. The treatment gave a maximum yield of 17·3% combined formaldehyde on rayon (cf. Wood's results [6], above), but only 5·8% of combined formaldehyde with cotton.

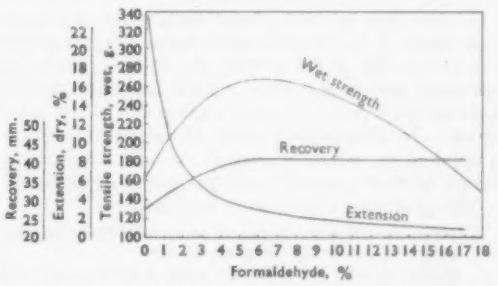


FIG. 1— Effect of Combined Formaldehyde on some Physical Properties of Viscose Rayon

Some of Saegusa's results were discussed in 1941 [13] and in 1946 [14], but the main graphs are now reproduced again in Fig. 1.

During the Second World War it was known "from information received" that considerable attention was being paid in Germany to the technical possibilities of formaldehyde-treated rayon and that goods were being produced containing 0·4–0·7% combined formaldehyde; for this and other reasons it was decided to re-examine this reaction, and some of the work is now described in the experimental section of this paper.

In 1941 it was found by Götze and Reiff [15] that the presence of formaldehyde reduced the degradation of rayon by acid; they also stated that the amount of combined formaldehyde increased with increase in concentration of acid catalyst during the reaction.

Dillenius [16], on the other hand, seemed to prefer a low concentration of catalyst and a high concentration of formaldehyde, presumably hoping to obviate acid hydrolysis of the cellulose.

Richter [17] showed that acid catalysts reduced not only the degree of polymerisation of cellulose in absence of formaldehyde but also the swelling value; it was therefore necessary to compromise between minimum degradation and maximum reduction of swelling. For this purpose ammonium chloride and aluminium chloride were stated to be superior to hydrochloric acid. Schaeffer [18] also stated a preference for aluminium chloride as catalyst.

In post-war years, Gagliardi and Grundfest [19] discussed crease-proofing in general, and in connection with formaldehyde treatments they showed that cotton was more susceptible than rayon both to improved recovery and to loss of strength. Kärrholm [20] confirmed Saegusa's observation regarding the physical effect on rayon of 5% combined formaldehyde and extended the maximum at this value to the moduli of bending, torsion, and stretching.

Woo, Dillon, and Dusenbury [21] carried out an extensive examination of the reaction between formaldehyde and cotton, rayon, and Fortisan, together with tests of the physical properties of the products; they confirmed the greater reactivity of rayon and the greater sensitivity of cotton in this process.

Experimental

In the first series of experiments a comparison was made of cotton and spun viscose rayon staple fabrics in the plain weave; the fabrics were not specially woven for these trials, but chosen only because their resistance to abrasion was of the same order. In the second series of experiments only a spun rayon fabric was used, and its weight was nearer to that normally encountered in dress goods.

The grey particulars were as follows—

Cotton S— 37·5 in. wide, 48 ends and 62 picks, 24s warp and 16s weft, 130 g./sq. metre

Rayon K— 38 in. wide, 49 ends and 46 picks, 14s warp and weft of 1·5 denier $1\frac{7}{16}$ in. staple Fibro, 174 g./sq. metre

Rayon T— 38 in. wide, 77 ends and 60 picks, 32s warp and 24s weft of Fibro as above, 117 g./sq. metre.

The fabrics were prepared in the bleachworks according to the usual procedure for such goods.

FORMALDEHYDE

The formaldehyde was obtained from Arthur Ashworth Ltd. of Bury; the formaldehyde content was 40·0% (wt./vol.) or 36·4% (wt./wt.) and the specific gravity was 1·099. It contained 5% of methyl alcohol and 0·015% of free acid.

ACIDITY

The only catalyst employed was tartaric acid; the pH of the various solutions was measured with the glass electrode firstly after neutralising with sodium hydroxide solution and secondly after adding the required amount of tartaric acid solution.

PLANT

The trials were conducted on a range normally employed for the production of crease-resisting goods, i.e. a three-bowl padding mangle followed by a pin-stenter in one operation, and a separate stage of heating in a festoon chamber having a temperature control within approx. $\pm 5°$C.

The goods were then washed in open width by passing through a series of compartments containing soap and sodium carbonate solution, warm water, and lastly cold water according to conventional methods; finally, the fabrics were dried on a pin-stenter.

TESTING

Analyses of combined formaldehyde were made according to the methods adopted by Wood [6], viz. by refluxing the samples with 20% ammonia for 30 min. to remove free formaldehyde, drying, hydrolysing with N. sulphuric acid containing sodium sulphate, and estimating the formaldehyde by the iodine method of Romijn [22].

Measurements of breaking load and extension at break were carried out on the Goodbrand machine, resistance to abrasion was observed on the ring-wear test apparatus [23], and crease-recovery was measured on the apparatus devised by Willows [24]. Water-imbibition, or water-retention, testing was carried out on the centrifuge originally used by Coward and Spencer [25] and having an acceleration of 2,900 ***g***.

Results

For various reasons the following results cover only part of the work done between 1941 and 1944; preliminary work on the effect of time, temperature, and formaldehyde concentration is not described, as it was only intended to establish conditions suitable for application to the works plant, and, in any event, similar work has since received detailed attention, particularly in the German literature.

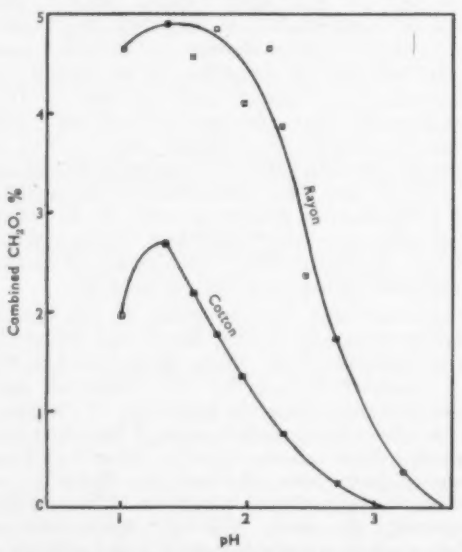


FIG. 2— Effect of pH on the Amount of Formaldehyde combined with Cotton and with Rayon (20% CH_2O soln. at 150°C. for 5 min.)

Furthermore, the experiments were directed to obtaining results within the range 0–5% combined formaldehyde only.

The results are limited to those obtained from heating the fabric at 150°C. for 5 min. after impregnation in 20% formaldehyde solution, generally at pH 1·7.

Fig. 2 shows the effect of pH on the amount of combined formaldehyde, and the results are in accordance with what might be expected from modern views of the relative proportions of amorphous material in cotton and rayon; they also agree with the reactivity of these two materials.

Even in view of the remarks about the limited range of the experiments, it may be well to emphasise that these results must not be taken to indicate that there are maxima of combined formaldehyde, roughly 5% for rayon and 2·5% for cotton; other work has shown that it is possible to obtain 17% of combined formaldehyde with rayon under different conditions.

The decrease in combined formaldehyde at approx. pH 1 is probably due to hydrolysis of the methylene ether; there is a similar effect with an excess of catalyst in the conventional crease-resisting process.

CREASE-RECOVERY

The crease-recovery of rayon and cotton as a function of combined formaldehyde is shown in Fig. 3. With both fibres the recovery reaches a figure of about 35–36 mm.; this is not necessarily the maximum recovery obtainable, but above these figures any further increase with increasing formaldehyde content is only slight.

For the same recovery, rayon requires about twice the amount of combined formaldehyde required by cotton; this is again in accordance with the relative amounts of amorphous cellulose, and if the combined formaldehyde is calculated on the amorphous part of the fibre, instead of on the whole fibre, the figure of approx. 8% is obtained.

It will also be seen that the response of cotton to improved recovery, resulting from combined formaldehyde, is more rapid than that of rayon, where the relation between recovery and combined formaldehyde is approximately linear.

If one compares the recovery effects of combined formaldehyde with those of added amino-aldehyde resins, the types of relation are very similar. For example, cotton responds more quickly than rayon, but when the gently sloping plateau is reached for both fibres, the added resin with rayon is about twice that for cotton, again in accordance with their amorphous cellulose contents. A comparison of the relation between dependent and independent variables, for both fibres and for both formaldehyde and resin, shows good correspondence for a weight of added resin which is three times the weight of combined formaldehyde.

STRENGTH AND EXTENSION

The breaking loads of rayon and cotton fabrics have been plotted against combined formaldehyde (Fig. 4). It will be seen that, in accordance with the results of previous workers, the wet breaking load of rayon rises rapidly, remains fairly constant, and then falls again; the results of Saegusa, for example, show the maximum wet strength at about 6% combined formaldehyde, with a decline at about 10% combined formaldehyde, whereas the data obtained by the author show a much earlier rise and fall. This may be due to the difference in the viscose rayons, or to the difference between continuous filament and spun rayon materials.

These possible differences may account also for the absence of any initial increase in dry tensile strength of rayon which has been reported by some

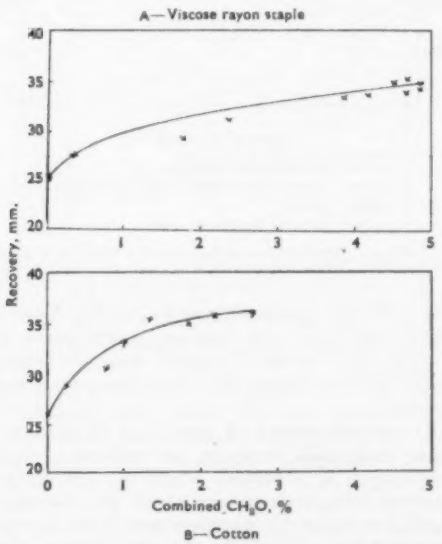


FIG. 3—Effect of Combined Formaldehyde on the Crease-recovery of Cotton and of Viscose Rayon (20% CH_2O soln. at 150°C. for 5 min.)

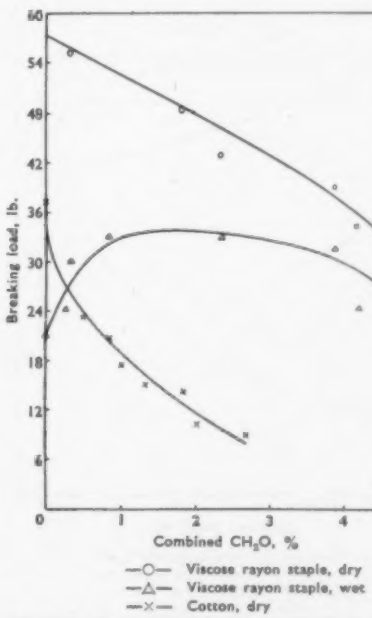


FIG. 4—Effect of Combined Formaldehyde on the Breaking Loads of Cotton and of Viscose Rayon (20% CH_2O soln. at 150°C. for 5 min.)

investigators[26]; however, all publications are agreed on the fall in dry tensile strength with increased quantities of combined formaldehyde.

The fall in breaking load of cotton is much greater than that of rayon, and even 1% combined formaldehyde is accompanied by 50% loss in strength. The reason for the great difference in behaviour between cotton and rayon is not fully understood, for it is well known that rayon is weaker and more reactive than cotton, so that one might have expected cotton to have suffered less from the treatment.

This type of reaction is complicated by the fact that the effects of acid hydrolysis accompany those of methylene ether formation; a second factor is the restriction of the reaction to the amorphous regions of the fibres. Because of the larger crystallites and larger proportion of crystalline material in cotton, the amorphous region is relatively more sensitive than that of rayon: the breakage of only a few chain-molecules by hydrolysis will bring about a greater fall in strength than the breakage of the same number of chain-molecules in rayon.

This greater sensitivity of cotton is not always realised, and sometimes causes surprise when it manifests itself; perhaps there is a need for a "sensitivity index" for cotton, mercerised cotton, rayon, and high-tenacity rayon.

An additional factor to be taken into consideration is that the formation of cellulose methylene ether under conditions of dehydration produces a reticulation effect which is not dissimilar from that produced by intensive drying, when there is a loss in the breaking load of cotton but a gain in that of rayon.

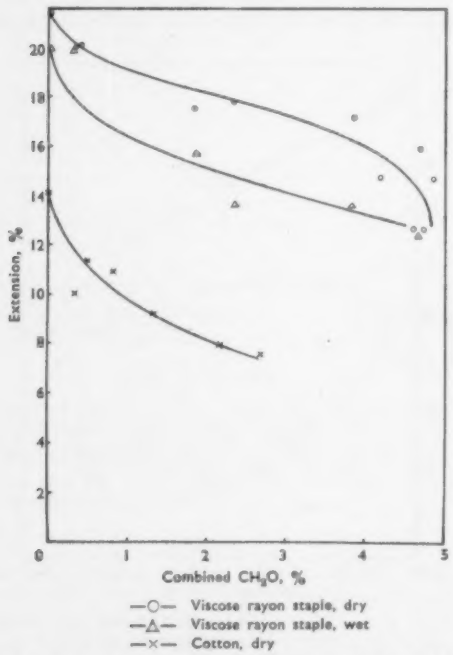


FIG. 5—Effect of Combined Formaldehyde on the Extension at Break of Cotton and of Viscose Rayon (20% CH_2O soln. at 150°C. for 5 min.)

Acid hydrolysis is not the whole story, however, for comparison of its effect alone with that of etherification in presence of formaldehyde shows that the formaldehyde increases the loss in strength with cotton, but decreases it with rayon.

The curves of Fig. 5 show a reduction in extension at break of rayon, in both dry and wet states, as the amount of combined formaldehyde increases. With cotton there is also a reduction in the extension at break with increase in combined formaldehyde, but the rate and the amount of fall are both greater than with rayon, as would be expected from cross-linkage of the molecular chains in smaller regions of poor lateral order.

RESISTANCE TO ABRASION

Tests of resistance to abrasion are not regarded with great confidence by many textile technologists, and there is considerable difference of opinion about the wisdom of accelerated destruction in general, apart from arguments as to the merits and demerits of particular forms of testing apparatus.

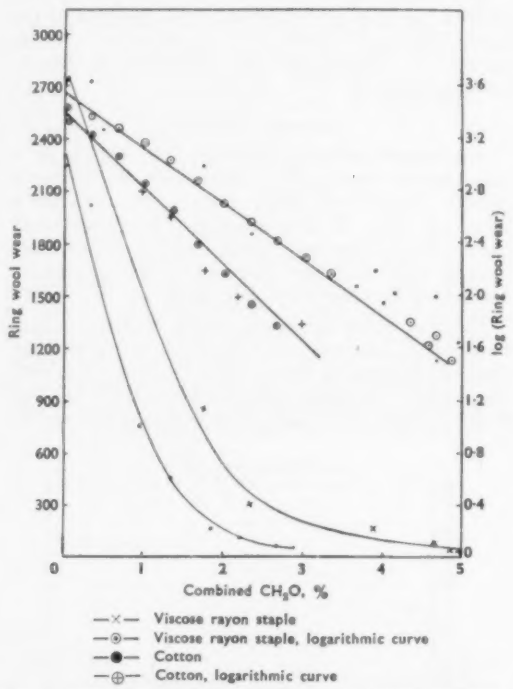


FIG. 6—Effect of Combined Formaldehyde on the Abrasion Resistance of Cotton and of Viscose Rayon (20% CH_2O soln. at 150°C. for 5 min.)

Nevertheless, the results set out in Fig. 6 are not without value, for first, they are comparative, and secondly, they show a regular relation between dependent and independent variables over a range of values.

The embrittlement of cotton is shown to be greater than that of rayon, but almost complete deterioration is observed when the amounts of combined formaldehyde are about 2% for cotton and 4% for rayon, i.e. in proportion to the amounts of amorphous cellulose in the two fibres. Comparing Fig. 6 with previous graphs, it is again

clear that for both fibres it is possible to obtain a satisfactory degree of crease-recovery only when it is accompanied by a most unsatisfactory amount of damage.

Spun Viscose Rayon Staple Fabric

A second series of experiments on a spun rayon staple fabric of lighter weight (117 g./sq. metre) confirmed some of the previous results and afforded the opportunity of extending others.

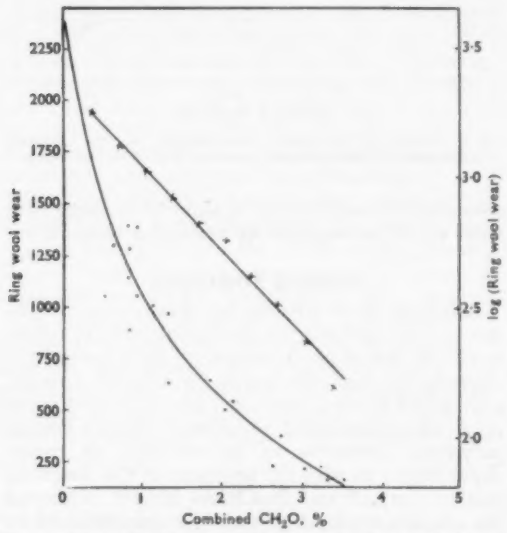


FIG. 7— Effect of Combined Formaldehyde on the Abrasion Resistance of Viscose Rayon Staple

The relation between resistance to abrasion and combined formaldehyde is shown in Fig. 7 and confirms the effects shown in Fig. 6.

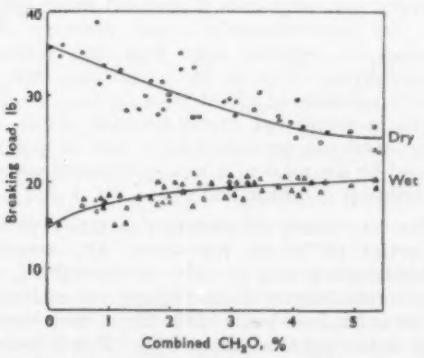


FIG. 8— Effect of Combined Formaldehyde on the Breaking Load of Viscose Rayon Staple

Breaking load, both dry and wet, is plotted against combined formaldehyde in Fig. 8. It will be seen that there is a greater irregularity with dry breaking load, which might be expected from a reaction which is rather difficult to control, not only on account of the competition between hydrolysis and etherification, but also because of the evaporation of formaldehyde during the reaction. It is for this reason that much work in Germany has been done with formaldehyde-donors.

At one time it was customary to express the wet strength of various fibres as a percentage of the dry strength as a basis of comparison; in the experimental results portrayed in Fig. 8 the wet: dry percentage appears to rise steadily from 42 to 80%. This is in agreement with some of the data of Saegusa [12], but with the higher amounts of combined formaldehyde which he obtained the wet: dry percentage rose to a maximum of nearly 90%.

WATER-IMBIBITION

The amount of imbibed water retained after centrifuging, and estimated as a percentage of the original weight, is shown plotted against combined formaldehyde in Fig. 9, a simple version of which was published in 1946 [27]. The relation between water-imbibition and combined formaldehyde appears similar to that between water-imbibition and added resin in the conventional crease-resisting process, and a comparison of the two sets of data is shown in Fig. 10; as with crease-recovery, there

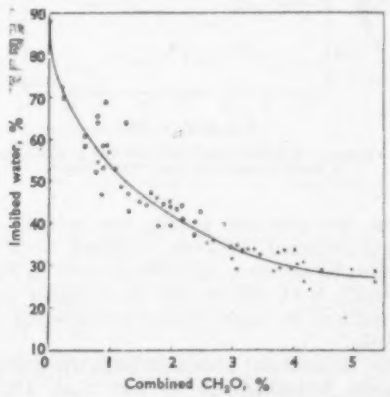
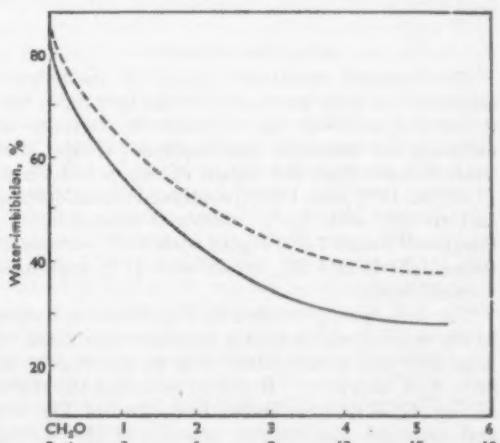


FIG. 9— Effect of Combined Formaldehyde on the Water-imbibition of Viscose Rayon Staple



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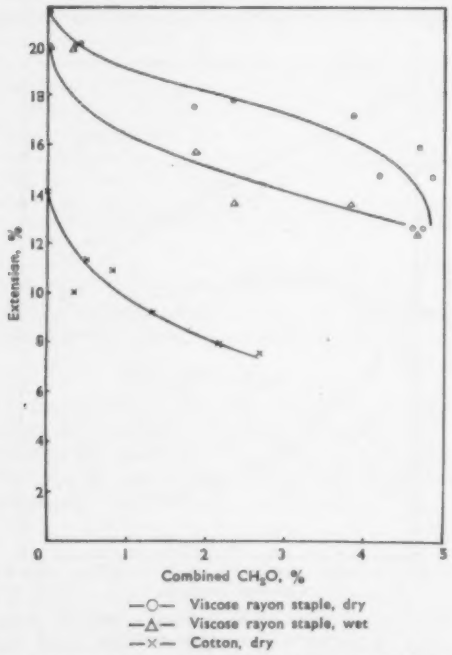

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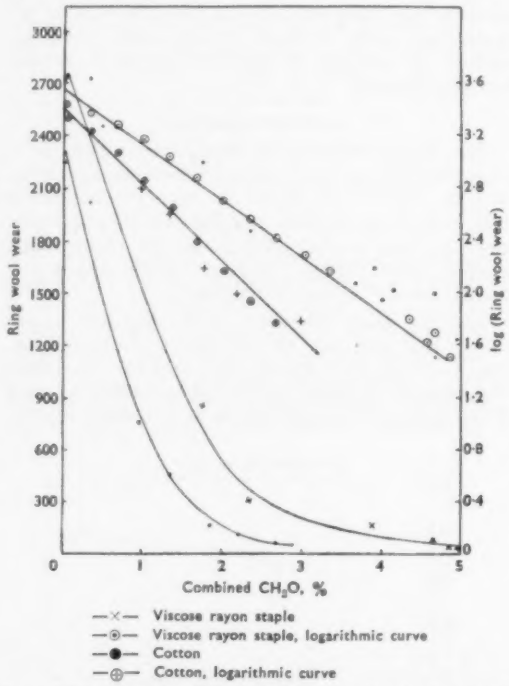

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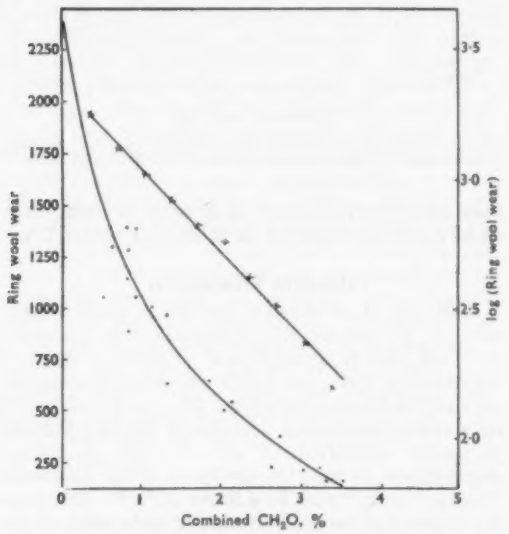

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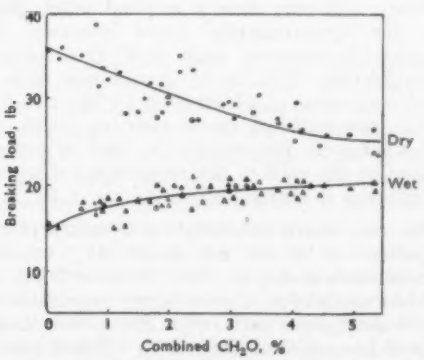

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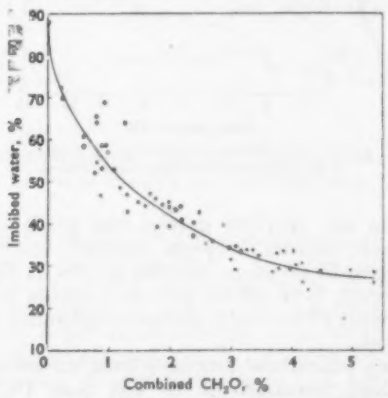

FIG. 9— Effect of Combined Formaldehyde on the Water-imbibition of Viscose Rayon Staple

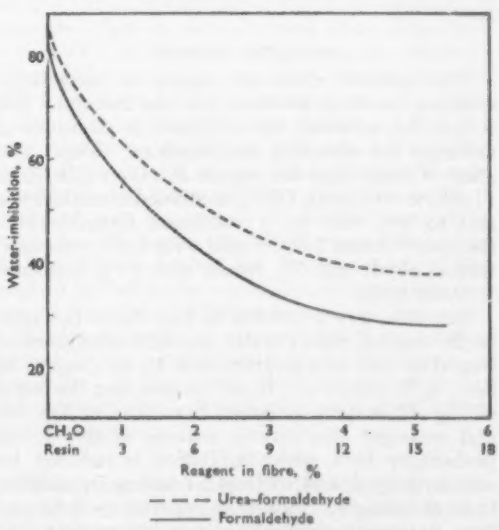

FIG. 10— Comparison of the Reduction in Water-imbibition of Rayon treated with Formaldehyde and with Urea–Formaldehyde

is a close relation between the effects of the two processes. Fig. 10 refers to a ratio of formaldehyde to urea of 1·6 : 1, and it is reasonable to assume that a still closer correspondence of the two curves would be found with a ratio of 2 : 1, but this has not been pursued.

When the logarithm of imbibed water is plotted against the logarithm of combined formaldehyde, many of the results lie on a straight line; linear relations are generally simpler to compare than curvilinear relations, but the log–log relation does not appear to have any practical value for determining required conditions in this particular reaction, as the results tend to be uneven.

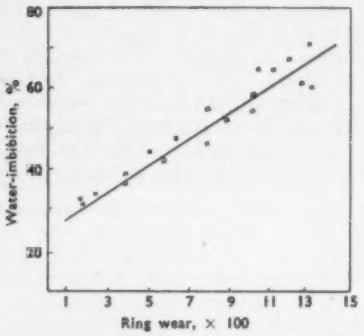

FIG. 11— Relation between Water-imbibition and Abrasion Resistance of Methylenecellulose from Viscose Rayon

From the previous graphs one would expect a linear relation between imbibed water and resistance to abrasion, and this is seen in Fig. 11. Apparently both effects vary in a similar manner as a result of the same, rather complicated, set of reactions.

Many commercial processes limit the amount of combined formaldehyde to less than 1%; this corresponds to a water-imbibition close to that which is often found with untreated cotton, viz. 50%.

MOISTURE REGAIN

The reduced moisture regain of methylenecellulose has been mentioned in the literature, but it has not received the emphasis it warrants as evidence for chemical combination. Goujet and Pinte [26] state that the regain of rayon falls from 11·5% to 10% with 1·25% combined formaldehyde and to 9% with 2·5% combined formaldehyde; Saegusa [12] found 7·5% regain with 3·3% combined formaldehyde and 6% regain with 17% combined formaldehyde.

The data now presented in Fig. 12 do not refer to the normal regain under standard conditions of humidity and temperature but to the regain at 95% R.H. and 25°c. It will be seen that the curve of Fig. 12 is quite different from that of Fig. 10, and amongst the various reasons is the strong probability that water-imbibition is reduced by acid hydrolysis and hydrogen bonding in addition to cross-linkages. Similar considerations deter one from drawing oversimplified conclusions from the ratio between the amount of resin and the

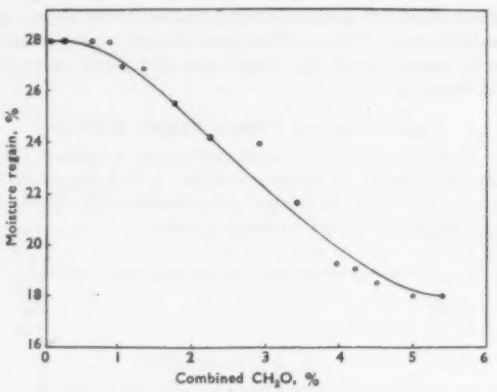

FIG. 12— Effect of Combined Formaldehyde on the Moisture Absorption of Viscose Rayon (regain at 95% R.H. and 25°c.)

amount of formaldehyde, in spite of its being near what could be regarded as molecular proportions.

General Discussion

Although it is possible to obtain good crease-recovery by forming the methylene ether of cotton or rayon, this is accompanied by such a degree of degradation that the goods are unmerchantable; anti-shrink finishes have been produced with rayon on a commercial scale, sometimes utilising various polymeric additives in an attempt to reduce degradation, as with the processes of the American Viscose Corpn.[28] and Dan River Mills [29]. Although the amount of combined formaldehyde required for anti-shrink effects is much lower than for crease-recovery, yet it is too much for the more sensitive cotton goods, which are severely damaged even at the lower level of combination.

Cotton is more responsive than rayon to the effect of the amount of combined formaldehyde on recovery: not only does it respond more quickly but, for approximately equal recovery from creasing, it requires only half the combined formaldehyde. This is in accordance with the relative amounts of added resin for the two fibres in the conventional crease-resisting finish with amino-aldehyde precondensates, and is probably decided by the ratio of the proportions of amorphous cellulose in cotton and rayon, viz. 1 : 2.

The very sharp deterioration in many physical properties of rayon at about 5% combined formaldehyde is seen in cotton at about 2·5% combined formaldehyde; if these figures are recalculated for the amorphous parts of the fibres, then they are more or less equal at about 8%. This is near the figure which one would expect (8·9%) from the substitution of one hydroxyl group per glucose residue by one part of the methylene bridge between the chain-molecules. However, it may be unwise to draw definite conclusions, for there may be polyoxymethylene cross-linkages—

$$-O\cdot CH_2\cdot[O\cdot CH_2]_n\cdot O\cdot CH_2\cdot O-$$

as well as the simple methylene linkage $-O\cdot CH_2\cdot O-$; indeed, it is very difficult to *prove* that cross-linkages have been formed, although there is a

great deal of evidence, some of which is accompanied by special pleading. Furthermore, there seems to be no reason for the regular substitution of one hydroxyl group in every glucose residue of chain molecules which are irregular in their disposition, and thus not prone to a regularity of cross-linkage.

One of the most remarkable properties conferred upon cellulose by reaction with formaldehyde is the insolubility in solvents such as cuprammonium; this takes place with as little as 0·1% combined formaldehyde, and comparisons have been drawn with the vulcanisation of rubber and the incorporation of divinylbenzene in vinyl polymers. Infrared spectroscopy, according to Lineken, Davis, and Jorgensen [30], has provided evidence that the reaction with formaldehyde reduces the number of hydroxyl groups in cellulose, and this is in accordance with analytical determinations.

The traditional belief in covalent cross-linkage of cellulose by formaldehyde owes much to constant re-asseveration of the views of Meunier and Guyot [5], which were based on measurements of water-retention after centrifuging, but there is other evidence to be considered. For example, it is now known that repeated drying or heating of cellulose will reduce the water-imbibition, presumably through hydrogen-bond formation, and it is less well known that hydrolysis also exerts an effect; the data of Richter [17] (cf. Table II) are often ignored.

TABLE II

Water-imbibition (%) of Rayon [17]

(3 hr. at 80°C.)

Catalyst (0·04 N.)	Formaldehyde 0%	Formaldehyde 5%
None ...	100	100
NH_4Cl ...	94	68
$(COOH)_2$ ...	85	65
H_2SO_4 ...	57	30
HCl ...	54	28

Part of the effect may be due to properties observed more recently, such as the recrystallisation mentioned by Ingersoll [31] with the accompanying fall in moisture regain noted by Howsmon [32]. Richter has also given some data for water-imbibition and degree of polymerisation produced by acid hydrolysis in absence of formaldehyde. It is obviously unwise to attribute the whole of the large reduction in water-imbibition to the cross-linkage, and to draw very close comparisons with the corresponding reduction in water-imbibition obtained with three times the quantity of urea–formaldehyde, where there is no hydrolysis of the chain molecules.

Insufficient importance has been attached to the fact that it is possible to combine formaldehyde with cellulose in presence of strong sulphuric acid, which acts as a swelling agent. The water-imbibition of the product is greater than that of the untreated material, so that reduced water-imbibition cannot always be taken as evidence of cross-linkage in cellulose.

Some of these high-swelling methylene ethers of cellulose exhibit crease-recovery in the wet state

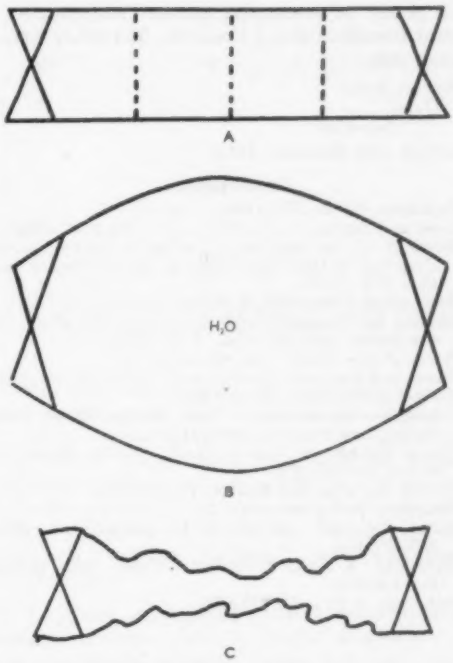

FIG. 13— Diagrammatic Representation of—
(A) Dry crease-recovery of collapsed fibre
(B) Wet crease-recovery of swollen fibre
(C) Absence of crease-recovery of (B) when dried.

only, and a similar effect has been developed with other cross-linking agents [33]. It seems probable that, where cellulose methylene ether is formed under conditions of dehydration resulting in a collapsed fibre, crease-recovery may be due to cross-linkages accompanied by hydrogen bonding, as illustrated diagrammatically in Fig. 13A; this might be expected to increase the rigidity of the fibre. Where reaction takes place on a highly swollen fibre, cross-linkages might be expected to keep the fibre in the distended state when wet, particularly where the swelling agent can penetrate the micelles; in the wet state there may be sufficient water-bonds to supplement the cross-linkage and increase the wet rigidity, so that the fibre resembles a canvas hose-pipe filled with water, as postulated by the writer among the early hypotheses for crease-recovery. The property of "wet but not dry recovery", which has recently attracted attention in the U.S.A., may be illustrated in Fig. 13B and 13C; the dry recovery of the product formed under conditions of strong dehydration is associated with synaeresis of the chain molecules, whereas the wet recovery of the product formed under conditions of strong hydration or dispersion is associated with diaeresis of the molecular chains, but both effects may result from cross-linkage.

* * *

Thanks are due to Mr. J. H. Butterworth for co-operation with the works trials, and to Messrs. N. F. Crowder and F. Pilling for assistance with the various tests. These investigations formed part

of a series of researches in the laboratories of Tootal Broadhurst Lee Co. Ltd., 56 Oxford Street, Manchester.

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(*Received 15th December 1958*)

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Setting Reduced Keratin Fibres with 1-Fluoro-2,4-dinitrobenzene

P. T. SPEAKMAN

Keratin fibres, when extended, reduced, and allowed to react with 1-fluoro-2,4-dinitrobenzene under either acid or alkaline conditions at 40°C., acquire a permanent set. Unreduced fibres and fibres reduced before extension are not permanently set. The effect may be caused by steric hindrance of mutual movement of protein chains by the large 2,4-dinitrophenyl group, or by hydrogen bonding between the nitro groups and ambient protein chains; or alternatively reaction with 1-fluoro-2,4-dinitrobenzene may alter the stability of the hydrogen-bonded protein system. The experiments seem to confirm earlier results which suggested that on extension certain cystine disulphide residues in the fibre become accessible to attacking reagents, and that these residues, previously inaccessible at 40°C., are those which are essentially involved in permanent setting.

INTRODUCTION

In 1941 Patterson *et al.*[1] showed that when a keratin fibre is reduced using thioglycollic acid the work necessary to extend the fibre 30% in water at 22°C. is lowered to 65% of the value for the fibre before reduction. If the cysteine residues formed during reduction are alkylated with methyl iodide the 30% index is only slightly increased (67%). If, however, heptyl bromide or benzyl chloride is used, the 30% index returns to a value close to that of the fibre before treatment (95% and 92% respectively), no doubt because the bulky hydrocarbon side-chain hinders movement of the protein chains.

Unreduced wool which has been allowed to react with 1-fluoro-2,4-dinitrobenzene (FDNB) in aqueous–alcoholic sodium bicarbonate solution to form the dinitrophenyl derivatives of the lysine and tyrosine residues, etc.[2] shows a decreased supercontraction in phenol[3], again because the bulky side-chain must hinder mutual movement of the protein chains.

Conventionally, permanent set is the percentage increase in length which remains after an extended and treated fibre is immersed without tension in distilled water at an arbitrary temperature (100°C.) for an arbitrary time (1 hr.). If, for example, a shorter time or a lower temperature were used, the set would be greater. Permanent set is thus an arbitrary measure of the stability given to keratin protein in the extended form. Forming side-chains on the protein chains of an extended fibre should make contraction more difficult and perhaps cause permanent set.

FDNB reacts with thiol and amine groups of model compounds at the pH of aqueous sodium bicarbonate, but in acetate buffers at pH values lower than 5·5 only thiol groups are dinitrophenylated[4]. In wool, however, some *O*- and *N*-dinitrophenylation takes place at the lower pH[5]. Also, it has been shown that under certain conditions FDNB can convert electrovalent salt linkages into covalent amide bonds[6].

RESULTS AND DISCUSSION

In the experiments described here, human hair, reduced while extended, has been allowed to react with FDNB above and below pH 5·5 at 40°C., and in both cases the fibres are permanently set if they are initially extended 45% (Fig. 1). Blank experiments (Fig. 2 and 3) show that dinitrophenylation of the lysine and tyrosine side-chains,

etc. of unreduced fibres is not sufficient to cause permanent set, and also that conversion of salt linkages to amide bonds cannot account for the effect. Residual set after immersing set fibres without tension for 1 hr. in boiling 5% aqueous sodium bisulphite solution shows that re-formed cystine disulphide cross-linkages are not responsible for the permanent set.

The absence of permanent set when unreduced fibres are used shows either that the thiol group is essential or that intact cystine disulphide bonds exert a restoring force on the protein system.

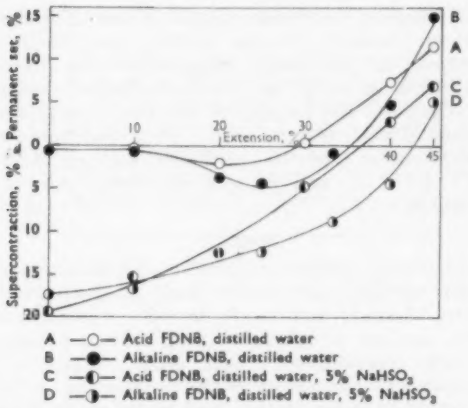

% Permanent set,
Supercontraction, %
Extension, %
A —○— Acid FDNB, distilled water
B —●— Alkaline FDNB, distilled water
C —◐— Acid FDNB, distilled water, 5% $NaHSO_3$
D —◑— Alkaline FDNB, distilled water, 5% $NaHSO_3$

FIG. 1—Fibres extended 0, 10....45%, reduced for 16 hr., immersed in FDNB at 40°C. for 2 hr., and then immersed without Tension in Boiling Water or Sodium Bisulphite for 1 hr.

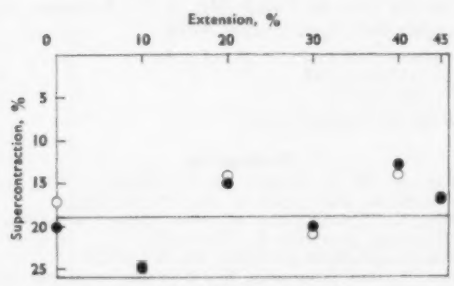


FIG. 2—Alkaline Buffer

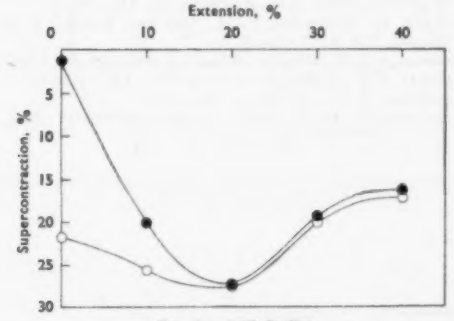


FIG. 3—Acid Buffer

FIG. 2 and 3—Fibres extended, reduced for 16 hr., immersed in Alkaline or Acid Buffer at 40°C. for 2 hr., and then immersed without Tension in Boiling Water or Sodium Bisulphite for 1 hr.

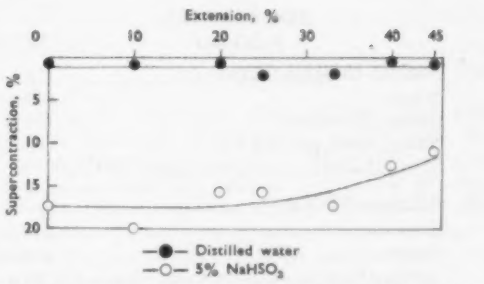
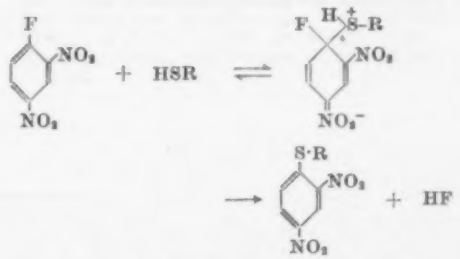


FIG. 4—Fibres reduced for 16 hr., then extended, immersed in Alkaline FDNB at 40°C. for 2 hr., and finally immersed without Tension in Boiling Water or Sodium Bisulphite for 1 hr.

Dinitrophenylation of lysine and tyrosine residues is not sufficient to cause permanent setting of unreduced fibres, in contrast to Zahn's experiment [3], which shows that it hinders the supercontraction of keratin fibres in phenol. It may be that bulky side-chains in the amorphous region of keratin, which seems likely to contain all the lysine and tyrosine residues [7], will not cause permanent set.

If the fibres are reduced *before* extension, even the more drastic, alkaline FDNB treatment of the subsequently extended fibres will not cause permanent set (Fig. 4). This suggests that there may be certain cystine disulphide groups, inaccessible to the reducing agent in unstretched keratin, which become accessible when the fibre is extended, and that reaction with these groups is essential for permanent set. Some earlier experiments using sulphite–bisulphite solutions as the setting agent also suggest this conclusion [7].

Thus the present results can be explained if the dinitrophenyl group, when attached to groups which are inaccessible in the unstretched fibre, gives a greater stability to the extended fibre, either by steric hindrance or by hydrogen bond formation with the nitro groups. Alternatively, Bunnett [8] has shown that the evidence from many bimolecular nucleophilic substitutions powerfully suggests intermediate complex formation. If this were the mechanism of the present reaction, the sulphur atom of the cysteine residue would acquire a positive charge in the complex. This charged atom might modify the hydrogen-bonded protein structure of the extended fibre and increase its stability [9]—



It is hoped that further work may make it possible to distinguish between these alternatives.

EXPERIMENTAL

Solutions

(*a*) Sodium thioglycollate—

Water	1,500 ml.
Sodium hydroxide	24 g.
Thioglycollic acid (84·9%)	50 ml.
Hydrochloric acid (conc.) to adjust pH to 4·6	

(*b*) Alkaline FDNB—

Ethyl alcohol	1,000 ml.
Water	500 ml.
Sodium bicarbonate (saturated) ...	20 g.
FDNB (measured molten)	20 ml.

(*c*) Acid FDNB—

Ethyl alcohol	1,000 ml.
Water	500 ml.
Sodium acetate (CH_3·COONa,3H_2O) ...	32·4 g.
FDNB	20 ml.
Hydrochloric acid (conc.) to adjust pH to 5·1	

(*d*) Alkaline buffer—
As (*b*) but without FDNB

(*e*) Acid buffer—
As (*c*) but without FDNB.

The pH of solutions (*b*) and (*d*) measured with a Cambridge pH meter and glass and calomel electrodes is 9·7, and of (*c*) and (*e*) 5·1. The significance of these measurements is obscure: Universal Indicator paper (BDH) shows solutions (*b*) and (*d*) to have a pH of approx. 8·0, (*c*) and (*e*) approx. 4·7.

Experimental Sequence

(i) Mount 4-cm. lengths cut from the root ends of purified human hair in individual setting frames, and measure their dry lengths with a travelling microscope. (ii) Extend the individual fibres 0%, 10%, . . . 45% in water, taking approx. 1 min. for the extension. (iii) Immerse the extended fibres in the sodium thioglycollate solution (*a*) at room temperature for 16 hr.[10]. (iv) Rinse in ethyl alcohol and distilled water, and immerse, still extended, in the FDNB solution (*b*) or (*c*) at 40°C. for 2 hr. (v) Rinse in ethyl alcohol and distilled water, and immerse without tension in boiling distilled water for 1 hr. (vi) Allow to dry, measure the length, and calculate the percentage increase or decrease (permanent set or supercontraction). (vii) Immerse without tension in boiling 5% sodium bisulphite for 1 hr., wash in distilled water, allow to dry, measure the length, and calculate the new permanent set or supercontraction.

Blank Experiments

(1) Experimental sequence as before, except that in step (iv) buffer (*d*) or (*e*) is used, with no FDNB. (2) As before, except that in step (iii) the fibre is immersed extended 45% in distilled water at room temperature for 16 hr., and not reduced. (The result is that the fibre contracts to 95% of its original length after 1 hr. without tension in boiling distilled water, and to 86·5% of its original length if the boiling-water treatment is followed by 1 hr. without tension in boiling 5% aqueous sodium bisulphite.)

Alternative Experimental Sequence

(i) Immerse the fibres mounted in setting frames without tension in the sodium thioglycollate solution (*a*) at room temperature for 16 hr. (ii) Rinse in ethyl alcohol and distilled water. (iii) Measure the lengths of the fibres (wet) and extend in water 0%, 10%, . . . 45%. (iv) Immerse the extended fibres in the FDNB solution (*a*) at 40°C. for 2 hr., and proceed with steps (v)–(viii) as before.

Reagents

Laboratory-grade thioglycollic acid standardised iodimetrically. Laboratory-grade sodium bisulphite, AnalaR sodium hydroxide, hydrochloric acid, sodium bicarbonate, and sodium acetate. FDNB recrystallised from ether; m.p. 25°C. Human hair purified by Soxhlet extraction: 12 hr. ether, 12 hr. ethyl alcohol, and rinsed many times in distilled water.

* * *

I would like to thank Mr. J. C. Atkinson for help with the experimental work.

DEPARTMENT OF TEXTILE INDUSTRIES
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(*Received 7th January 1959*)

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CORRESPONDENCE

The Editor does not hold himself responsible for opinions expressed by correspondents

The Relation between Molecular Weight and Diffusion Coefficient in Dyeing

The diffusion of dyes into fibres and films has been studied by many workers. In some cases where a range of dyes has been examined [1–3], an inverse correlation has been observed between the molecular weight of a dye (regarded as an approximate measure of molecular size) and its diffusion coefficient in the substrate. No attempt has been made to express this relationship in a quantitative manner.

The conditions under which such a relationship might be expected are— (*a*) when the pores in the fibre are not much larger than the width of the dye molecule, and (*b*) when the forces of attraction between dye and fibre are the same for a range of dyes. With cellulosic fibres the pores are relatively large, and rate of diffusion is not governed by the size of the dye molecule. On the other hand, with disperse dyes and polyethylene terephthalate the type of attraction appears to differ with varying chemical constitution, and no correlation is found between diffusion coefficient and molecular size [4].

A study of the literature has shown two cases [2,3] where the relationship can be expressed by the empirical equation—

$$\log D = a + kM^{\frac{1}{3}}$$

where D is the diffusion coefficient, M is the molecular weight, and a and k are constants. This is illustrated in Fig. 1 for disperse dyes in secondary cellulose acetate film at 80°C.[2], and in Fig. 2 for acid dyes in a block of Ultramid 6A (a copolymer of 6-nylon and 6,6-nylon) at 80°C.[3].

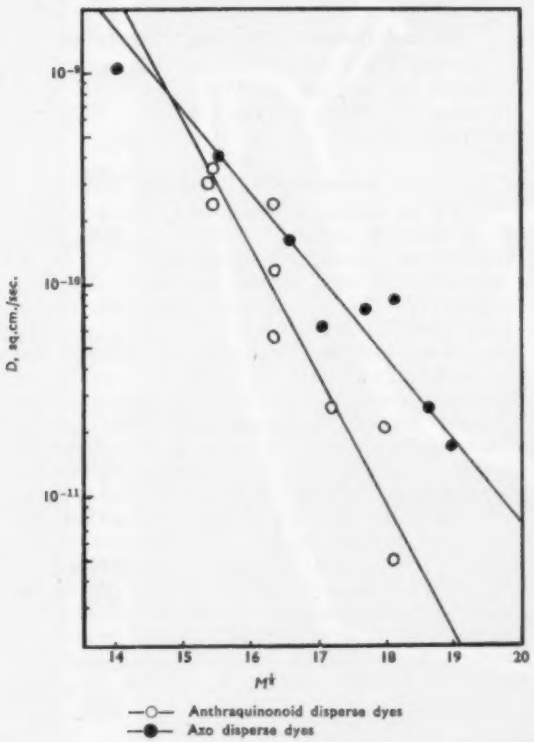

FIG. 1— Correlation between log D and $M^{\frac{1}{3}}$ in the Dyeing of Secondary Cellulose Acetate at 80°C.

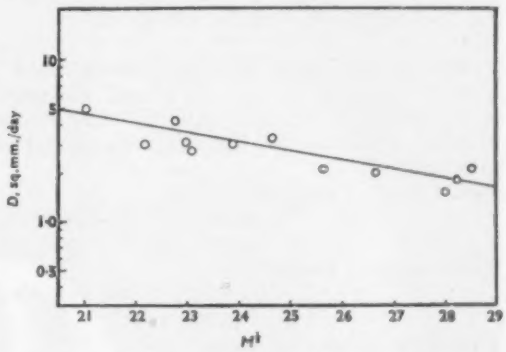

FIG. 2— Correlation between log D and $M^{\frac{1}{3}}$ in the Dyeing of Ultramid 6A at 80°C.

It is seen (Fig. 1) that the azo disperse dyes diffuse more rapidly than the anthraquinonoid dyes as the molecular weight increases above about 240, possibly owing to the different molecular shapes of the two types of dye.

I would like to thank Mr. C. L. Bird, of the University of Leeds, for his kind assistance.

K. ODAJIMA

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Notes

Meetings of Council and Committees
April

Annual General Meeting— 10th

Council— No meeting

Finance and General Purposes— 1st

Publications— 21st

Diplomas— 17th and 30th

International Federation 1959 Congress— Social Subcommittee— 20th

International Relations— 22nd

Burnt-gas Fumes Fastness Subcommittee— 8th

Annual Meeting of Chairmen and Honorary Secretaries of Sections— 10th.

Deaths

We regret to report the deaths of Dr. E. Higgins, Mr. F. L. Jones, and Mr. E. Young.

West Riding Junior Members Competition

Prizes awarded by the Dyers and Finishers' Association have been won by the following Junior Members of the Society for essays on the topics indicated.

First Prize — Roy Seddon
The Colour and Constitution of the Triphenylmethane Dyes

Second Prize — Frank Wood
The Chemistry of the Diazotisation Process

Third Prize divided between
Michael Dunsmore
The Chemistry of the Diazotisation Process
and
Brian Nestor
Preparation and Properties of Fibrous Polyamides

The prizes were presented to the successful candidates by the President of the Dyers and Finishers' Association (Mr. L. Morton Wood) at the Annual General Meeting of the West Riding Section of the Society, held at the Victoria Hotel, Bradford, on 9th April 1959.

Scottish Section Annual Essay Prize

Last year the Committee of the Scottish Section decided to institute a prize, in the form of books to the value of £3 3s. 0d., to be competed for by members of the Scottish Junior Branch. The subject chosen for the 1958–1959 session was "Invention and the Dyeing Industry", and the prize was won by Mr. Ian Fleming for his essay entitled *Phthalocyanine Dyes*. The prize was formally presented at the Annual General Meeting of the Scottish Section held at the St. Enoch Hotel, Glasgow, on 10th March 1959.

John Thompson Marsh

Mr. J. T. Marsh, M.Sc., F.R.I.C., F.T.I., F.S.D.C., has been elected an Honorary Life Member of the Textile Institute.

Terylene Patents

The Patent Office announced on 13th March 1959 that the term of the Terylene patents would be extended for five years. Since then an appeal has been lodged.

Oil and Colour Chemists' Association

The Oil and Colour Chemists' Association has changed its address to Wax Chandlers' Hall, Gresham Street, London E.C.2 (Telephone MONarch 1439).

Xerographic Printing

A talk on *Recent Developments in Xerography* by K. A. Metcalfe and R. J. Wright, to which members of the Society of Dyers and Colourists of Australia had been invited, is very briefly summarised in *J. Oil & Col. Chem. Assocn.*, **42**, 105–106 (Jan. 1959). Xerography is a "dry" photographic process in which the light-sensitive plate consists of an electrically charged layer of a photoconducting insulating material on a normally conducting base, so that the optical image is converted into an electrostatic image. Among new xerographic techniques demonstrated were colour printing and overprinting (both with and without visible light), printing on metal, and paint analysis, and coloured survey maps were displayed. The speakers claimed that textiles could be printed by inducing an image in the cloth, or alternatively by coating the fabric, printing, and finally dissolving the intervening coating layer. Moisture would tend to aid rather than hinder xerographic textile printing, provided that the moisture content did not exceed 50% by weight.

New Books and Publications

Determination of Fastness to Daylight of Coloured Textiles
Amendment No. 1 to B.S. 1006:1955

PD 3078. Pp. 3. London: British Standards Institution. June 1958. No price.

This amendment lists *Colour Index* designations for the dyes employed for the blue light-fastness standards, modifies the angle and other details of exposure, and states that, if two different assessments are obtained at the two different degrees of contrast, then their arithmetic mean shall be taken as the light fastness. A new appendix contains a discussion of the use of fading lamps. The substance of this amendment, other than the interim report on xenon arc lamps, has already appeared in this *Journal*.

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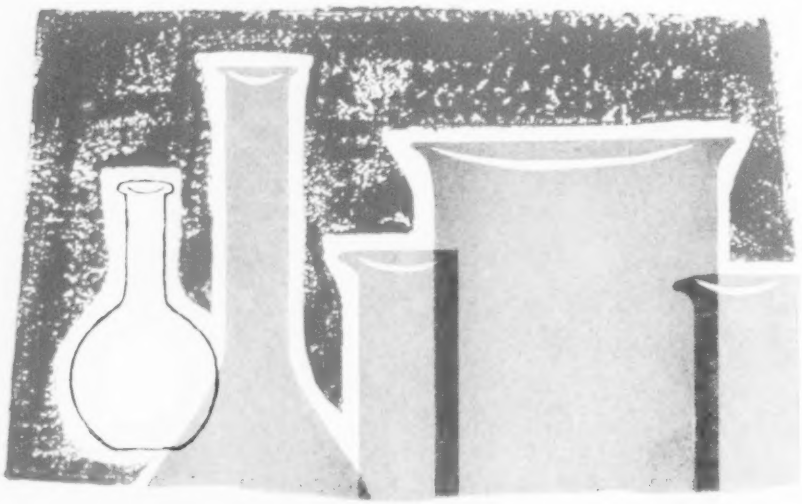

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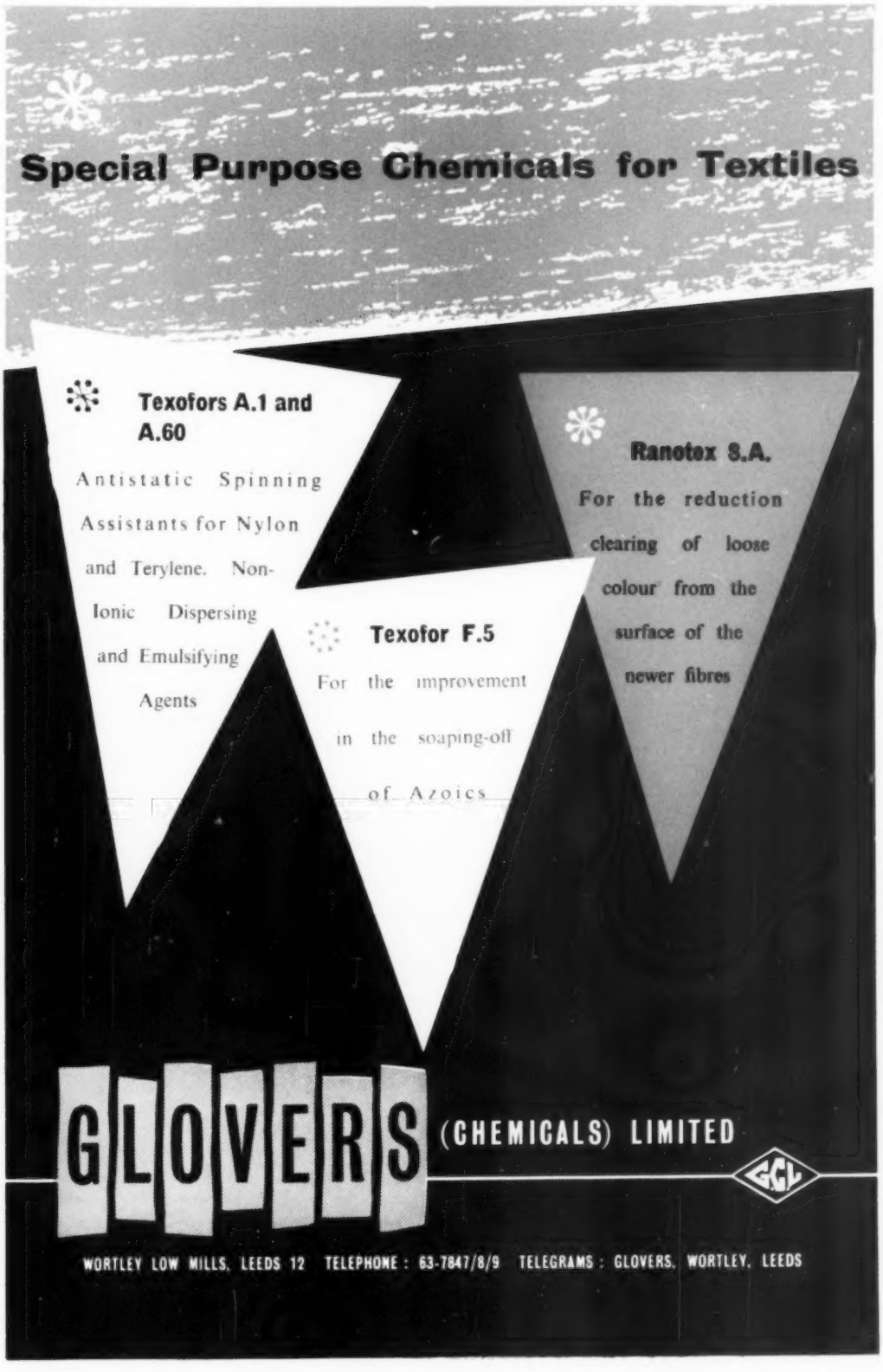
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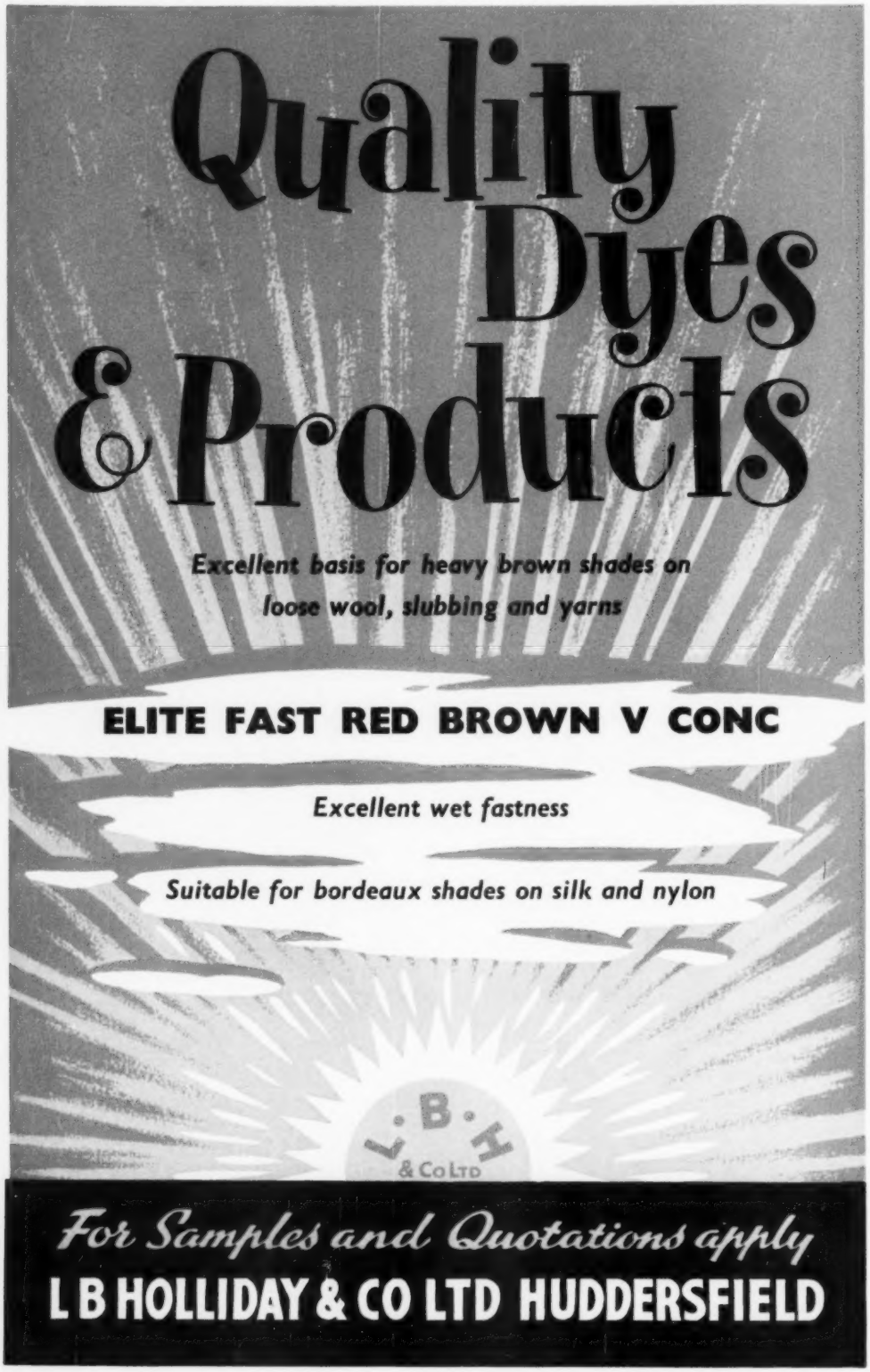
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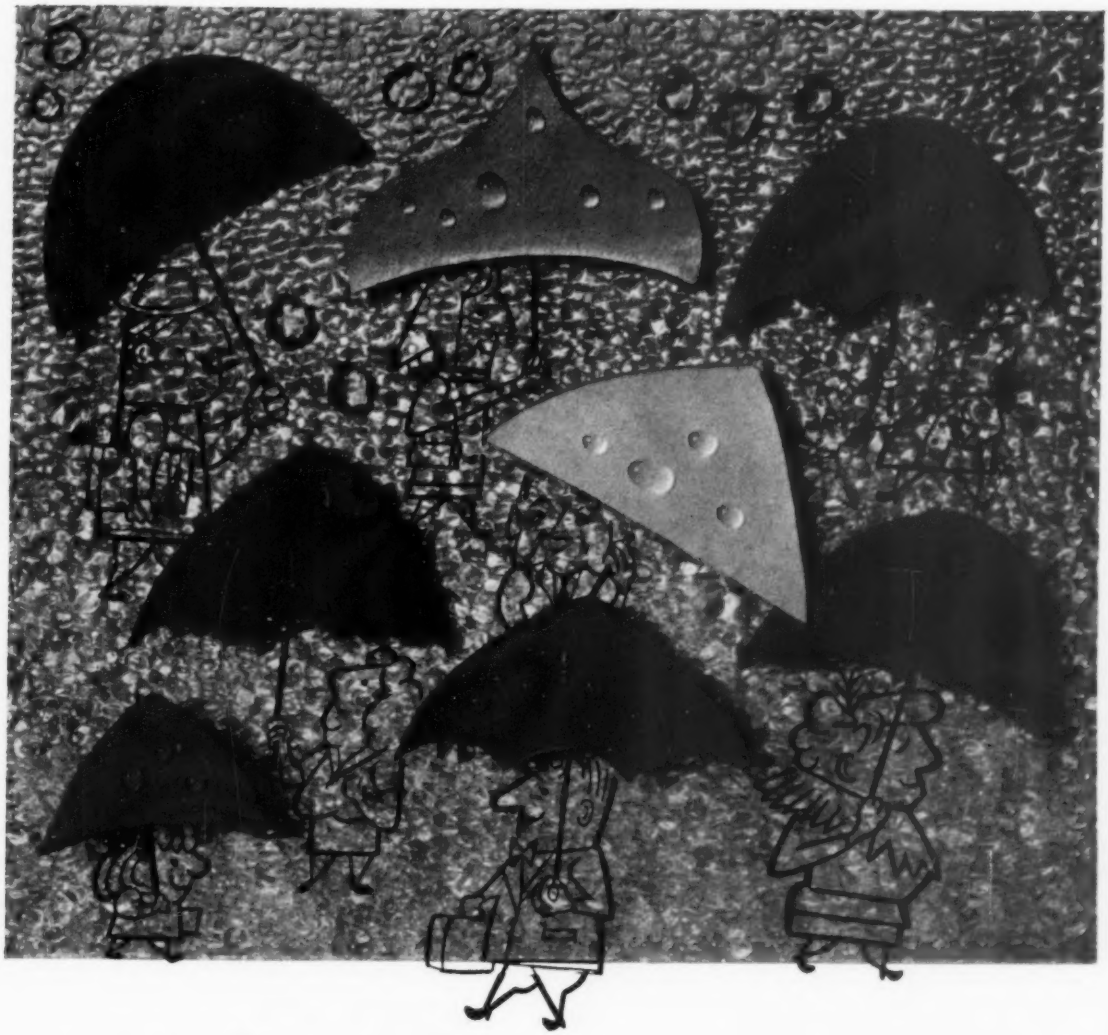

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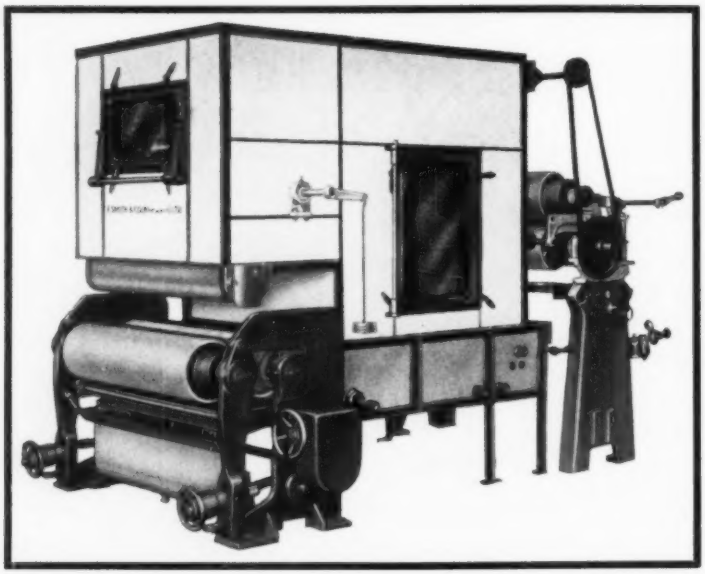

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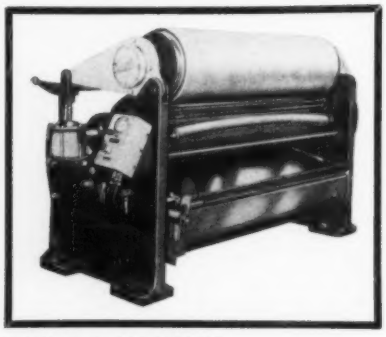

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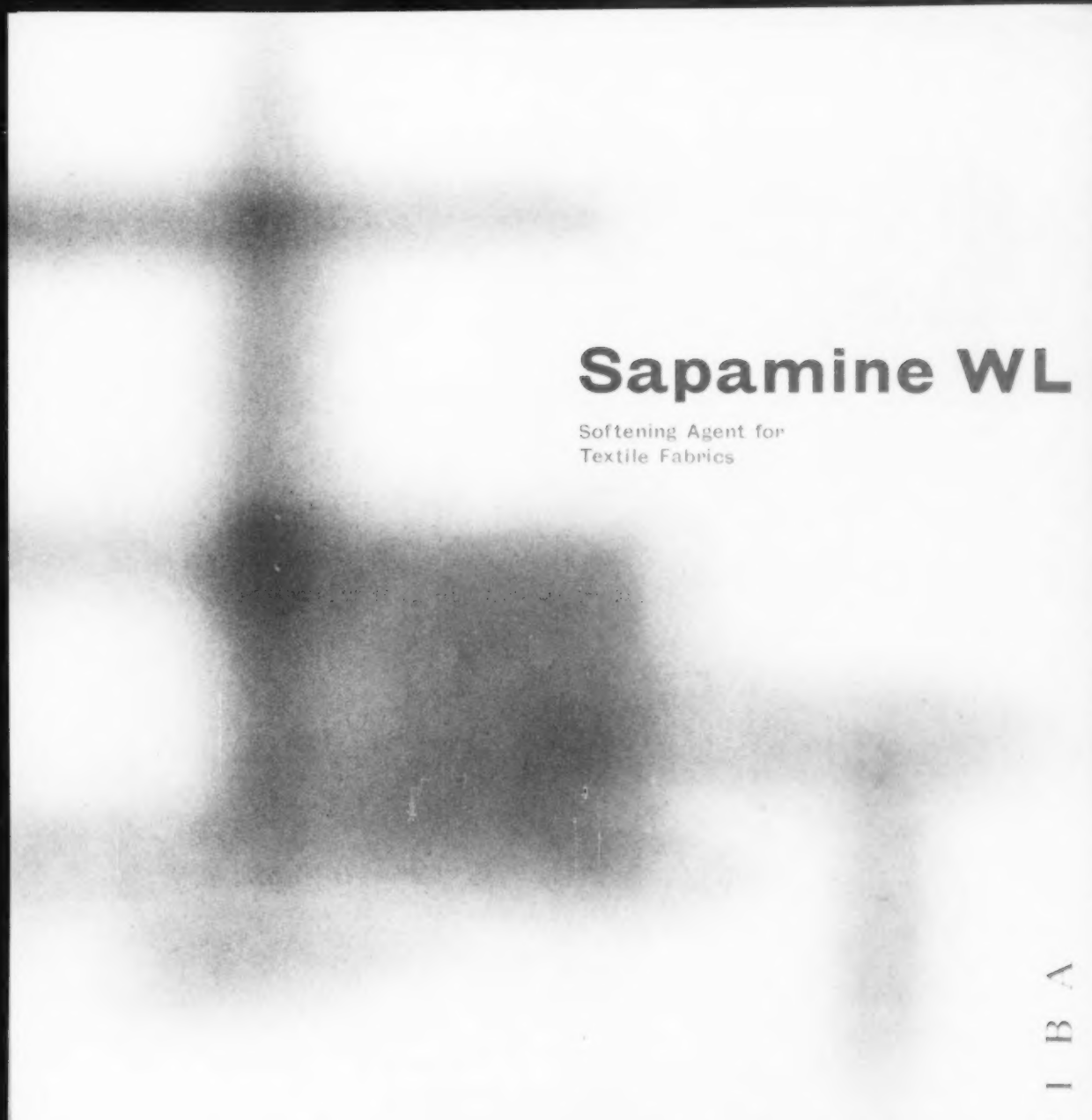

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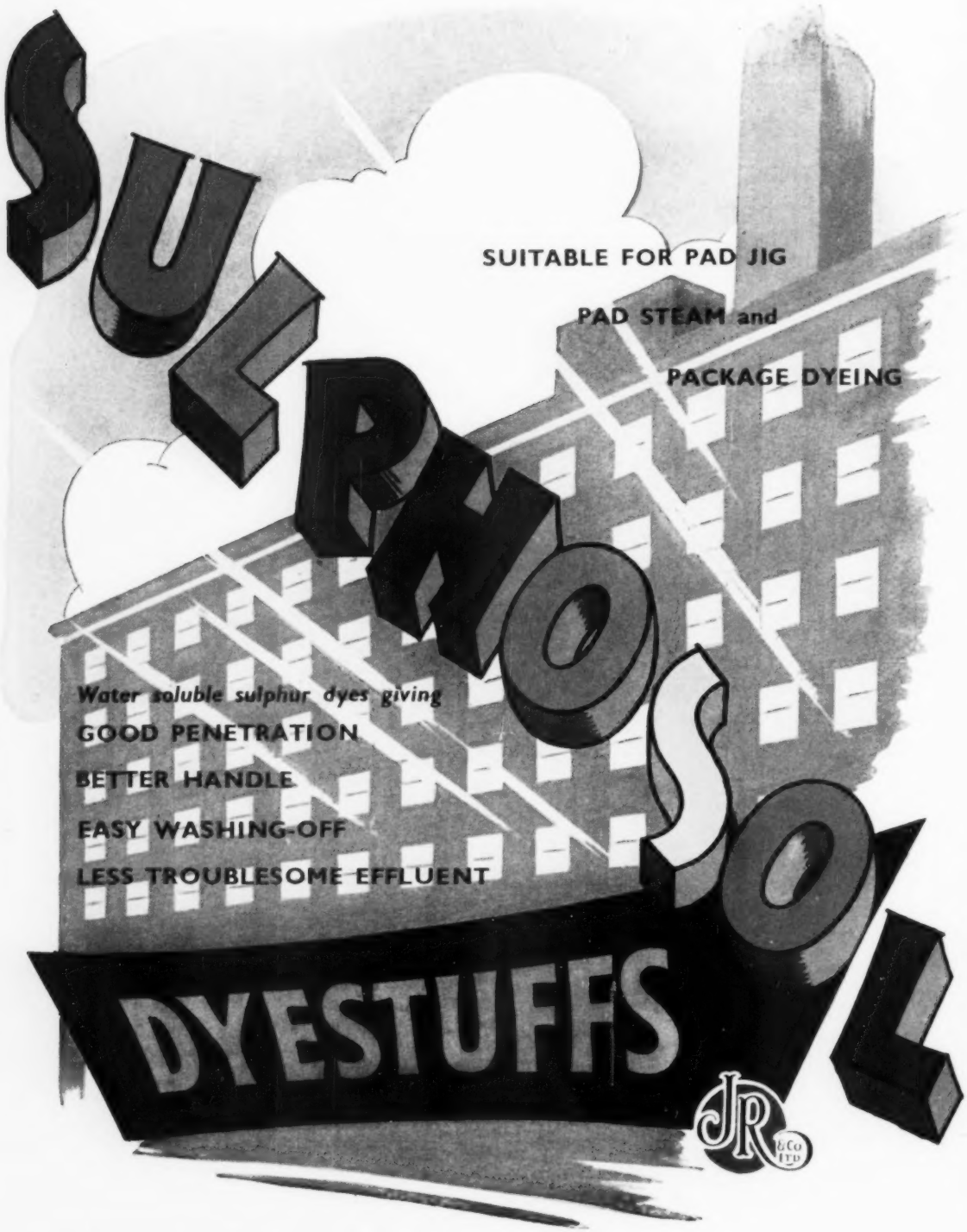

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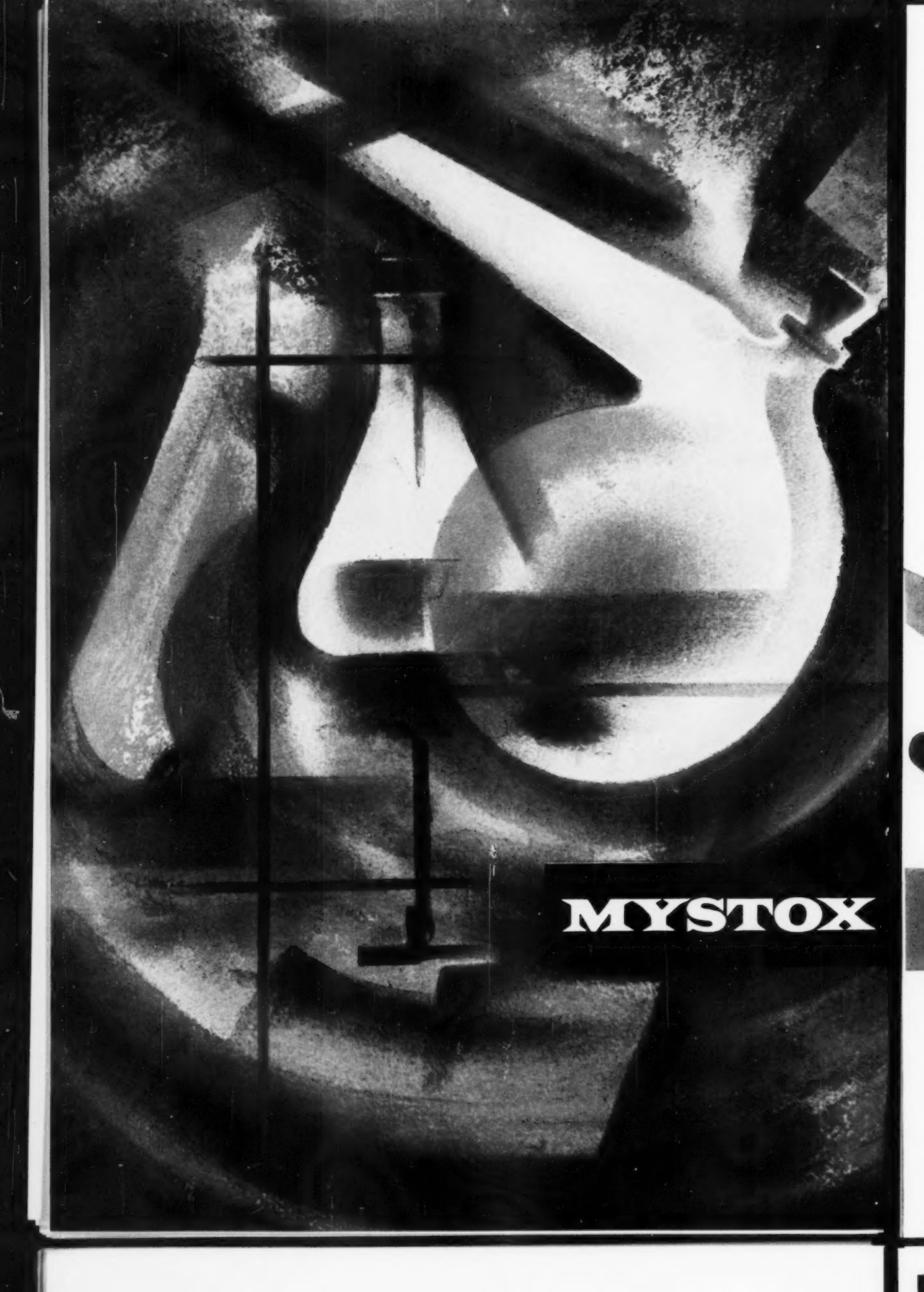
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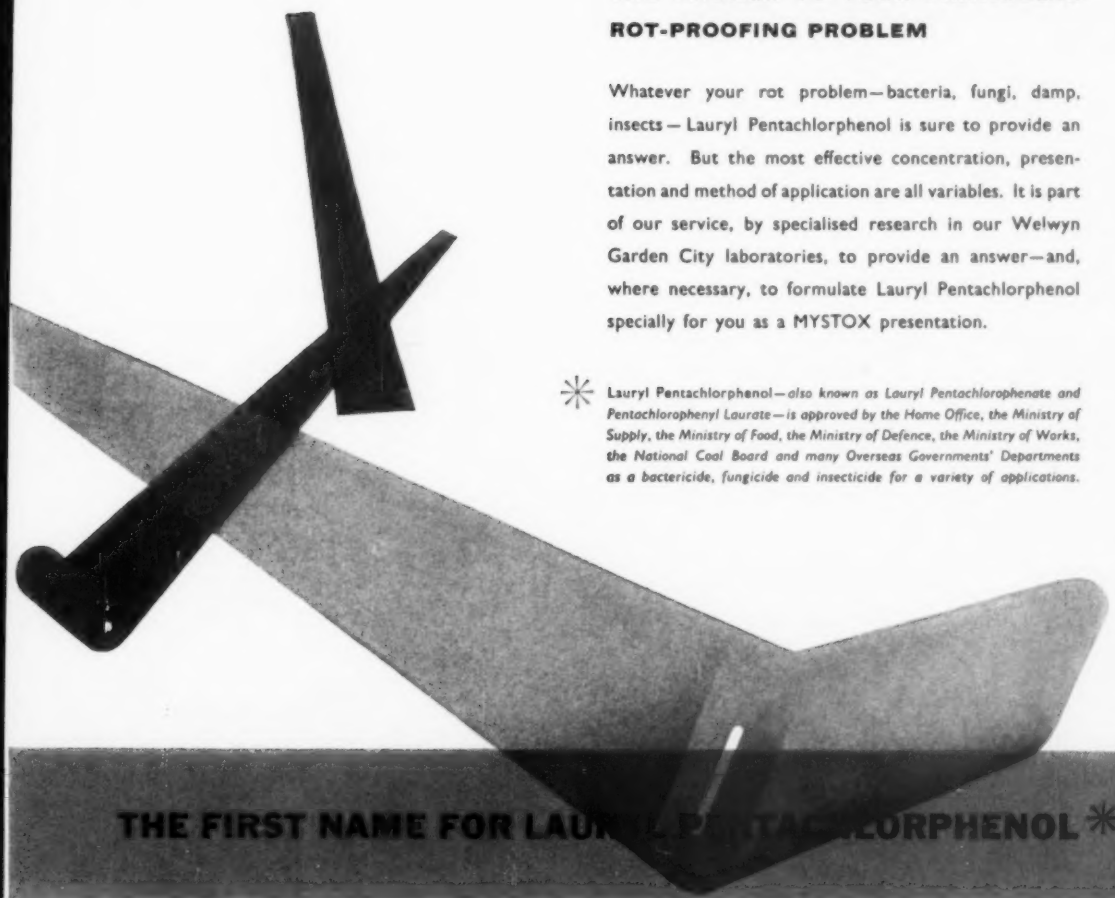

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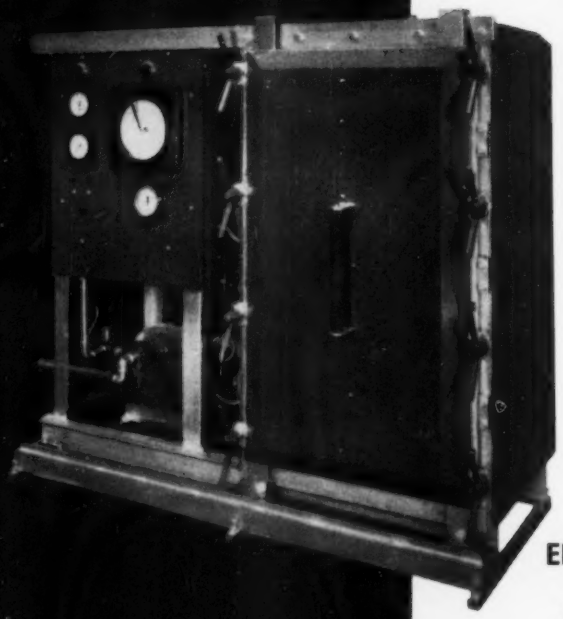

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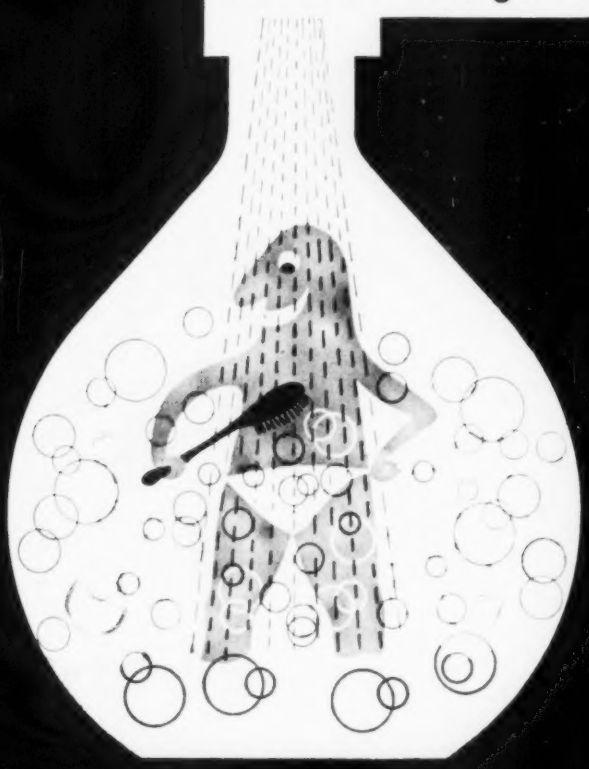

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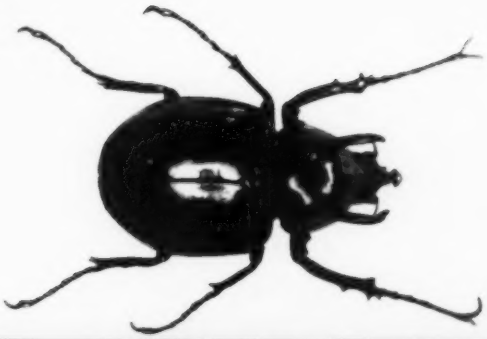

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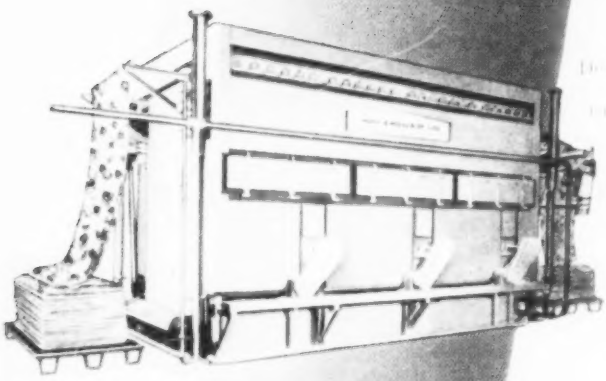

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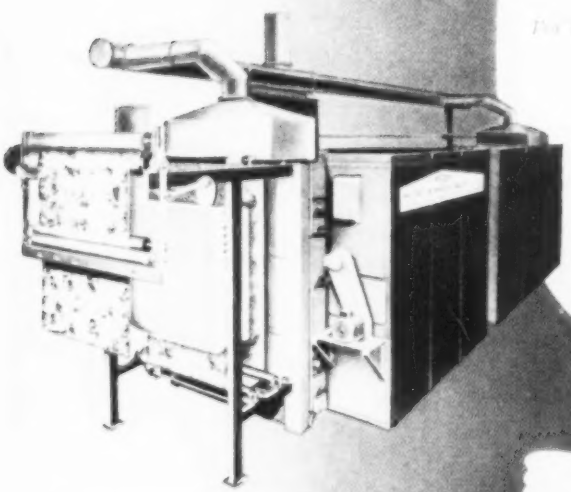

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Bradford Textile Society Journal 1957–58

Bradford. Pp. 136. Price, 10s. 0d.

This issue maintains the stimulating appearance and the general interest that we have come to expect from the Bradford Textile Society during recent years, for which the Honorary Editor, Mr. G. G. Hopkinson, the printers, and the block-makers deserve every credit. As usual, there is an array of articles and illustrations more or less related to the wool trade, dealing especially with historical and geographical aspects and with current affairs. However, this issue contains rather less material than usual directly concerned with coloration and finishing. E. A. Busby, from the retailer's point of view, discusses the increase in popularity of dyed and printed cottons for dresses, the pointlessness of detailed washing and cleaning instructions, and the dangers of publicity not based on a well tried product. W. H. Bower's description of blanket manufacture includes some mention of dyeing and finishing.

C.J.W.H.

The Man-made Fibres Industry

By R. Robson. Pp. viii + 135. London: Macmillan & Co. Ltd. (New York: St. Martin's Press Inc.). 1958. Price, 21s. 0d.

The author deems the time now opportune to present in book form information on the economic development and structure of the man-made fibres industry previously scattered in official statistics and the technical and trade press.

Chapter I, on *Processes*, discusses the various ways of classifying man-made fibres before going on to outline the methods of manufacture of the various regenerated and synthetic-polymer fibres.

Chapter II, on *Development*, traces the growth of the industry from Comte de Chardonnet's patent for "nitrocellulose silk" in 1885 to the latest dinitrile fibre. It is interesting to see how each new development, after passing through periods of teething troubles and of rapid expansion, flourishes, and then tends to lose ground to a newer development, unless it is saved by finding a fresh outlet. Thus by 1904 annual production by Chardonnet's process had reached 2 million lb., but by 1913 even the original Chardonnet plant had changed to the more efficient viscose process. Rayon was used at first in braids and embroideries, and then in hosiery, before being taken up by cotton weavers. During the 1930s acetate rayon filament began to encroach on viscose rayon especially in the fine yarns, but the expansion of the latter was maintained by the development of staple and of high-tenacity filament for tyres, both substitutes for cotton. Later, blends of viscose rayon staple were spun both on the cotton and on the woollen and worsted systems. Since the Second World War nylon filament has almost completely conquered the market for ladies' hose, and is beginning to replace rayon in tyre cord. Synthetic staple is replacing rayon in the wool industry, and polyester and polyacrylic fibres are tending to gain on the polyamide and polyvinyl fibres. However, developments in mass coloration, bonded-fibre fabrics, and tufted carpets are providing other new outlets for rayon.

Chapter III, on *Structure*, is of considerable interest in showing how the interplay of the traditions and necessities of the textile and chemical industries has led to the conditions of today: relatively few giant firms dealing especially with the chemical processes, where efficiency is favoured by large-scale operation, and a larger number of smaller firms with the mechanical processes, which can be carried on efficiently on a much smaller scale. In some cases integration has proceeded backwards to machinery design and raw materials, e.g. lumber for rayon, and forward to textile processing, e.g. weaving and dyeing.

Chapter IV, on *Prices and Costs of Production*, makes clear to the layman some of the difficulties encountered in attempting to compare the economics of the different fibres. Thus the cost of chemicals may not reflect their real cost of production, since it may be determined by the demand from other sources (e.g. acetic anhydride for cellulose acetate) or the demand for a "co-product" of the process, which applies particularly among petroleum chemicals.

Chapter V, on *Natural Fibres and other Competing Materials*, shows that, here again, comparative costing encounters difficulties, in some cases because different fibres tend to meet different needs. It is rather surprising to learn on p. 93 that, for the same tenacity, rayon and nylon cost about the same, with steel twice this cost and cotton midway between the two.

The last chapter, devoted to *Fibre Properties and Inter-fibre Competition*, stresses that textiles are in competition with other materials, e.g. paper and plastic sheeting, for certain purposes, and that in many cases there is a shifting competition between different fibres and blends, affected by changes in fashion and taste, so that in recent years research has been directed especially towards the requirements for different end-uses of textiles.

Five appendixes outline the scientific background, dealing with fibre structure and yarn structure, polymerisation, wood pulp and cellulose, raw materials, and basic chemicals and monomers respectively.

Altogether this book provides a very valuable and interesting conspectus of the industry which has provided the dyer, the printer, and the finisher with a large proportion of their headaches during the twentieth century. It would have been improved by rather more careful editing; e.g. words are occasionally misspelled ("hydrophylic", "liquifies"), and in a book published in 1958 it is intriguing to read about capacity planned for 1957 (p. 36).

C.J.W.H.

Japanese Textiles

By Tomoyuki Yamanobe, English adaptation by Lynn Katoh. Pp. ii + 70 + frontispiece + 7 interleaved colour plates. Rutland (Vermont) and Tokyo: Charles E. Tuttle Co. (*Arts & Crafts of Japan* No. 2). 1957. Price, $3.00.

Although the main emphasis of this little book is on handicrafts rather than technical matters, the profuse illustrations (a total of 21 colour

plates and 37 plates in gravure and halftone) and descriptions of a tradition of colourful textile design quite different from our own contain much of interest to dyers and printers. Dyeing and weaving in Japan seem to date from about the first century B.C., and by the Asuka–Nara period (A.D. 552–794) dyeing included several tie-and-dye methods and a kind of batik or wax dyeing, a silk gauze dyed by a clamped-stencil technique being illustrated. Later, in the Muromachi period (1336–1568), the Tsujigahana style was evolved, in which tie-and-dye methods were used to produce hand-painted designs. Yuzen dyeing, the forerunner of modern screen printing, was developed in the seventeenth century; the technique, in which rice paste is used as a resist, is briefly described.

Informative notes are given on the illustrations, which are mainly of kimonos and other textiles preserved in various museums in Japan. For one design "dozens of stencils" were employed; another resembles at first glance a series of formulae of polynuclear vat dyes. A satin Noh costume contained warp threads "dyed in even lengths of white, red, purple, and light green".

The book concludes with a list of 22 Japanese natural dyes, illustrated by a colour chart, and a glossary of 46 terms, including seven related to dyeing.

C.J.W.H.

Oral Communication of Technical Information

By Robert S. Casey. Pp. vii + 199. New York: Reinhold Book Division (London: Chapman & Hall Ltd.). 1958. Price, $4.50 (36s. 0d.).

The author regards this book as a "do it yourself" manual for technical people who wish to speak effectively, especially in public. He claims to have collected many helpful suggestions from the widely scattered articles on the subject, extensive bibliographies to which are given at the end of each chapter, on forms of "oral communication" ranging from ordinary conversation to the formal presentation of papers. However, it is difficult to judge how far much of this information is really useful to would-be speakers or is merely a rather naïve labouring of the obvious interspersed with counsels of perfection. On the other hand, many of the hints concerned with the practical organisation of meetings appear to be very valuable, and are rarely stated explicitly in print.

The first five chapters (pp. 1–68), concerned with importance and scope, barriers and bridges, methods of working, organisation, and composition, say a great deal that is equally applicable to written communication, and are interesting for the sometimes amusing sidelights apparently thrown on American conditions. A useful warning about the dangers of misunderstanding due to the often unconscious prejudice of the listener is illustrated by a story about a flop house— without quotes. On p. 50 dictionaries surprisingly appear to be preferred to usage as a guide to the correct meanings of words, although in fact they only record existing usage. On the same page the reader will be confused by the alleged examples of unfortunate connotations, for they depend rather on the existence of homonyms. The author is even more severe than the Fowlers in stating (p. 56) that "while" and "since" should be used only for temporal relationship, but later (p. 72) he feels it necessary to warn his reader against such pronunciations as "bronichal", "lahrnyx", and "drownded".

Chapters 6 and 7 (pp. 69–97) are concerned with the delivery of formal and informal talks. Oral delivery requires a more colloquial and natural style than a written report, and also careful consideration of the outlook of the listeners. It is interesting that the range of acceptable rates of speaking is 90–160 words a minute. Pauses but not hesitation are necessary to replace the punctuation of the written language. These chapters contain also some salutary comments on mannerisms and other distractions, talking to the screen, reading slides, timing, and rounding off numbers.

Chapter 8, on mechanical techniques and audio and visual aids, is the longest and appears to the reviewer to be the most useful in the book. Among the more elaborate aids are— wireless microphones, allowing mobility to the speaker; remote operation of slides by the speaker; an overhead projector enabling the speaker to project even chemical reactions, to point, and to write while still facing his audience; magnetic display boards; and the TelePrompTer. The section on "Standards and Specifications for Visual Aids" is extremely valuable, recommendations being given both for the preparation of copy (graphs, tables, photographs) for slides and for the brightness and size of the projected image to ensure maximum comprehension and legibility. The rest of the chapter is devoted to the preparation of slides, models, and exhibits.

Chapter 9 contains some pertinent advice on the chairmanship of meetings and the organisation of symposia, include a wiring diagram for a signalling system enabling the chairman to indicate to the speaker when his time is up. The last three chapters are concerned with talking science to laymen, technical legal testimony, and social conversation, this last being contrasted with the communication of information.

All those who have to speak at meetings or who are concerned in their organisation will find some valuable ideas in this little book.

C.J.W.H.

New Books received

Recent Progress in the Chemistry of Dyes and Pigments. W. Bradley. (Lectures, Monographs, and Reports 1958. No. 5). London: Royal Institute of Chemistry. Pp. ii + 94. 9s. 0d.

Steric Effects in Conjugated Systems. Chemical Society. (Proceedings of a symposium at Hull University). London: Butterworths Scientific Publications. 1958. Pp. viii + 181. 30s. 0d.

1959 Year Book. Philadelphia: Federation of Paint and Varnish Production Clubs. Pp. 175. $2.50.

Handbuch für Textilingenieure und Textilpraktiker. Teil T13— Mikroskopie der Faserstoffe. Paul-August Koch. Wuppertal-Elberfeld, Germany: Dr. Spohr-Verlag. Sixth edition. 1959. Pp. viii + 97. DM 7.50.

Handbuch für Textilingenieure und Textilpraktiker. Teil T15— Textilchemische Prüfungen. PAUL-AUGUST KOCH. Wuppertal-Elberfeld, Germany: Dr. Spohr-Verlag. Sixth edition. 1959. Pp. vi + 142. DM 10.00.

Chemische Technologie und Praxis der Färberei. Band II— *Die Farbstoffe und das Färben der Zellulosefasern, synthetischen Fasern und Fasergemische.* GERHARD NITSCHKE. Berlin: VEB Verlag Technik. 1958. Pp. 451. DM 36.00.

Analytical Applications of Diamino-Ethane-Tetra-Acetic Acid. T. S. WEST and A. S. SYKES. Poole, Dorset: British Drug Houses Ltd. [1959]. Pp. 106. 4s. 6d.

Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., **68**, *23 (Jan. 1952) and also, together with symbols and the periodicals abstracted, in the annual index*

I— PLANT; MACHINERY; BUILDINGS

Crushing and Grinding

A Bibliography. London, H.M. Stationery Office, 1958. Pp. 425. Price 35s. 0d.

Pigment Dispersion in Szegvari Attritor

CDIC Paint and Varnish Production Club Technical Committee (H. L. Fenburr, Chairman)

Off. Dig. Fed. Paint Varn. Prod. Cl., **30**, 1280–1284 (Nov. 1958)

The Szegvari Attritor is a laboratory-scale mill in which one gallon (U.S.A.) of flint pebbles (ca. ¼ in. diameter) are impelled by a vertical shaft having six arms perpendicular to the shaft and moving at 160–200 r.p.m. The mill tank has a capacity of ca. 1¼ gal. (U.S.A.). The speed of dispersion depends on composition and appears to be optimal at the composition corresponding to the Daniel pour point. Complete dispersion of TiO_2 in a short-oil alkyd is obtainable in as little as 10 min. J.W.D.

Continuous Dyeing Machinery for all Dye Classes

R. B. Vogel and H. Hölters

Melliand Textilber., **39**, 1243–1246 (Nov. 1958): 1365–1368 (Dec. 1958)

All cotton dyes can be applied in a plant comprising the sequence— padding mangle, hot-flue, air passage, padding mangle, steamer, air passage, open soaping range— provided that the fabric is uniformly dry and absorbent. Up-to-date versions of the requisite machinery, singly and in combination, are described, and the requirements, of the following dye classes are discussed in detail— vat, direct, reactive, leuco vat ester, azoic, and sulphur. 17 illustrations. S.M.J.

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Heating Cloth by Direct Contact with Steam

DuP *USP* 2,858,184

Steam is passed into the upper part of a storage vessel, e.g. a J-box into which cloth impregnated with a treating liquor is continuously passed and temporarily stored. The steam is introduced into direct contact with the cloth at a point above which the fabric is stored as a compact mass, and it flows countercurrent to the cloth. C.O.C.

Rope or Open Width Washers

Joh. Kleinewefers Söhne *BP* 810,049

J-shaped washing chambers are placed in series and the fabric in either rope or open width is passed through them so that it is stacked in each chamber. The fabric is passed through at a fairly high speed but in spite of this the duration of the action of the liquid on the fabric is considerably lengthened because of the stacking up of the fabric in each chamber. The scouring liquid flows countercurrent to the fabric. This arrangement enables the water consumption to be reduced to a minimum by controlling the amount of material in each chamber and the flow of liquid in relation to the speed of the fabric. C.O.C.

Dyeing Machine Employing Vibrational Energy

L. S. Adams Engineering Co. *USP* 2,858,689

A torsionally-operated roll is situated beneath the level of the dye liquor. About this roll is placed a series of parallel pressure rollers which press the fabric against the torsionally operating roll as this roll moves the fabric in open width through the liquor with an oscillating motion. This results in the fabric being subjected to high frequency squeezes which force the dye liquor in and out of the fabric. C.O.C.

Polymerising Resins in Travelling Cloth or Paper

Spooner Dryer & Engineering Co. *BP* 809,821

The impregnated material is led into apparatus where it is first heated to near the temperature needed for polymerisation by directing onto it either perpendicularly or at a large angle to it high velocity streams of hot gas. Polymerisation is then carried through to completion while the hot material is surrounded by an atmosphere of slowly-moving hot gas. C.O.C.

Silk Screen Printing Frame

G. O. Leibenguth *USP* 2,854,922

A frame in which the stencil fabric is so mounted that the tension imparted to it can be adjusted and so that the stencil is readily removed and replaced. C.O.C.

Drying Impregnated Porous Webs

Kimberly-Clark Corpn. *USP* 2,838,420

Impregnated porous cellulose fibre webs, such as resin-impregnated crêpe cellulosic wadding and similar, are dried by passing a stream of air or gas to and fro through the web, reversing the direction of the flow several times before the impregnant is immobilised. In this way migration of the resin is prevented. R.A.

Humidifying Hydrophobic Materials

American Viscose Corpn. *BP* 809,694

The material is passed either vertically or horizontally through a chamber where it is treated with saturated steam. The supply of steam is either reduced or cut off when passage of the material temporarily ceases. It is specially suitable for the conditioning of cellulose film and glassine after it has been dried during printing. C.O.C.

New Products developed since November 1957— Alphabetical List of Colouring Materials, Chemicals, and Equipment (IV p. 261)

Screen Printing (IX p. 271)

Impregnated Fibre Webs (XI p. 273)

III— CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Continuous Production of Detergents

W. S. Fedor, B. Strain, L. Theoharous, and D. D. Whyte

Ind. Eng. Chem., **51**, 13–18 (Jan. 1959)

A description is given of the plant and continuous process methods developed and used by the Procter & Gamble Co. for the manufacture of fatty alcohol sulphates and dodecylbenzenesulphonate. W.K.R.

Catalysts in the Sulphation of Castor Oil

S. Rangarajan and N. P. Palaniappan

Ind. Eng. Chem., **50**, 1787–1788 (Dec. 1958)

The sulphation of castor oil with 25% by wt. of 92% sulphuric acid was studied at 29·5°C. in the presence of catalysts (1% on wt. of acid). Mercury and mercurous sulphate gave 40–50% higher yields and increased the rate of reaction, but vanadium pentoxide and copper sulphate had little effect, and pyridine acted as a negative catalyst. W.K.R.

Production of Terephthalic Acid

II— Liquid-phase Air Oxidation of *p*-Xylene under Pressure and Liquid-phase Air Oxidation of Methyl *p*-Toluate

N. Ohta and T. Tezuka

Rept. Govt. Chem. Ind. Research Inst. Tokyo, **53**, 271–276 (xxx–xxxi) (Aug. 1958)

III— Liquid-phase Air Oxidation of *p*-Cymene and Di-isopropylbenzenes

N. Ohta *Ibid.*, 343–350 (xl–xli) (Oct. 1958)

PATENTS

Detergent
Henkel–Helios AB. *BP* 810,151

A detergent whose aqueous solution at < 50°C. is at pH 7·5–9·5 and after some time at 80–100°C. rises to 9·4–11·0 is made up of a nonionic or anion detergent (but not a soap), an alkali metal phosphate and MgO, the latter being present as a powder whose individual particles are coated with a substance melting at 50–60°C., e.g. cetyl alcohol. C.O.C.

Bleaching Composition
Whiffen & Sons *BP* 810,128

A stable very powerful bleaching composition comprises hexachloromelamine mixed with a solid filler and/or buffer compound, e.g. a mixture of hexachloromelamine (1–15%), monosodium phosphate (20–60) and sodium sulphate (79–25). C.O.C.

Sequestration of Ferric Iron in Caustic Alkali Solution
Norman Evans & Rais *BP* 810,113

Ferric iron in caustic alkali solution is completely sequestered by use of an alkylolamine and/or a polyalkylolamine. Thus dihydroxyethylaminediacetic acid is fully effective to pH 8·5 and retains 50% of its efficiency at pH 9·7, while hydroxyethylethylenediamine triacetic acid remains effective at pH 11·5. C.O.C.

Antistatic Agents
BASF *BP* 810,352

Quaternary ammonium compounds of polymers or copolymers of *N*-heterocyclic compounds whose vinyl group is directly attached to a C or N atom of the heterocyclic nucleus, e.g. the polyquaternary ammonium salt of *N*-vinylimidazole and dimethylsulphate, are excellent antistatic agents for undyed fibres, aqueous solutions or emulsions of 0·1–0·5% being effective. The antistatic finish imparted has comparatively good fastness to washing. C.O.C.

Size for Yarns
ICI *BP* 809,745

The stable aqueous dispersion obtained by polymerisation in presence of a persulphate catalyst of acrylic or methacrylic esters in an aqueous solution of a polysaccharide, the weight of polysaccharide being 10–300% of that of the ester, is used as a textile size. C.O.C.

Size
Monsanto *USP* 2,859,189

An aqueous size consisting of an ammonium salt of a vinylacetate–crotonic acid copolymer and a little stearic or other fatty acid of 12–20 C, permanently sizes regenerated cellulose or cellulose acetate filaments. The sized filaments show no tendency to stick to the drying cans. Fabrics woven from the sized yarn do not water spot and may be printed without being desized. C.O.C.

Anhydrous Textile Finishes
American Viscose Corpn. *USP* 2,857,330

Addition of a compound of formula—

$$R\dot{N}\text{—}(CH_2)_y\text{—}\dot{N}\cdot(CH_2\cdot CH_2\cdot O)_xH$$
$$(CH_2\cdot CH_2\cdot O)_xH \quad (CH_2\cdot CH_2O)_xH$$

(R = aliphatic hydrocarbon of 8–18 C; x = 1–3; y = 2–4) inhibits textile finishes formed by dispersing the ingredients in mineral oil in absence of water, from corroding machinery. Thus to Ethoduomeen T/13 (2 parts) and glycerol trioleate dispersed in Textiline 85 (a highly refined low-viscosity mineral oil) (68) was added soya ethyl morpholiniumethosulphate (2 parts). Applied to acetate yarn passing over a roller so that there was a pick-up of 3%, no corrosion occurred during two weeks of continuous use. The treated yarn was soft, non-electrostatic and well lubricated. C.O.C.

Emulsions of Hardenable Resins
Ciba *BP* 809,504

Aqueous emulsion of solutions of water-insoluble hardenable aminoplast resins in an alcohol of limited miscibility with water, e.g. butyl alcohol, and made by use of a non-ionic emulsifying agent, are stabilised by addition of a water-insoluble, liquid aliphatic hydrocarbon, e.g. petroleum of boiling range 150–260°C. If desired the emulsion may be concentrated by evaporation. C.O.C.

Stable Aqueous Solutions of Amino-aldehyde Resins
Monsanto Canada *BP* 809,662

Stable aqueous solutions of modified melamine–formaldehyde resins are obtained by reacting together in aqueous medium 1 mole of melamine with 2·5–6·0 moles of formaldehyde and enough water-soluble polyhydric alcohols, containing > 1 primary hydroxyl group, e.g. ethyleneglycol, to give the ratio of 0·2–1·0 primary hydroxyl group per hydroxymethyl group of the melamine–formaldehyde addition product. The reaction is continued until one volume of the resin solution is precipitable by < 2 and > 4 vol. of water at 25°C. The amount of reactants used is such as to yield a 50–60% resin solution. The resulting solution is stored at pH 8·5–9·5. C.O.C.

Catalyst for Organosiloxane Finishes
Midland Silicones *BP* 809,766

A mixture of Zr and Zn acetates in which Zr : Zn :: 0·1–4·5 : 1 can be added to organohydrogenosiloxane emulsions without affecting their stability. When such an emulsion is applied to cloth and cured at 95°C. for 10 min., immediate water repellency is obtained without the need for ageing. C.O.C.

Cold Permanent Waving Lotion
Gillette Co. *BP* 810,355

An aqueous solution of 0·4–0·9 M. of thioglycolic acid, thiomalic acid or dimercapto adipic acid containing 0·8–1·4 M. of a mixture of ammonia or a monoalkylolamine and a dialkylolamine is effective for waving hair which is otherwise difficult to wave, e.g. damaged or bleached hair. The amount of primary and secondary amine is each 0·4–0·9 M. and the total free base present is 0·2–0·6 M. C.O.C.

Flame-resisting Agent
U.S. Secretary of Agriculture *USP* 2,859,134

Compounds containing > 1 1-aziridinyl group attached to pentavalent P atoms react with amino compounds to yield polymers containing the recurring connecting structures—

$$R\cdot\dot{N}\cdot CH_2\cdot CH_2\cdot\dot{N}\cdot P \quad \text{and} \quad P\cdot\dot{N}\cdot CH_2\cdot CH_2\cdot\dot{N}R$$

(NR = an amino radical). Deposited on hydrophilic fibrous organic materials they reduce the combustibility and are fast to laundering. They can also be deposited on the surfaces of non-hydrophilic materials to form flame-resistant coatings. Thus cotton cloth was padded with an aqueous solution containing tris(1-aziridinyl)phosphine oxide and ethylene diamine at pH 12·1, dried for 15 min. at 90°C. and baked for 5 min. at 140°C. The treated cloth contained 13·4% resin, was highly flame-resistant, and remained flame-resistant even after being boiled for 3 hr. with soap and soda. The handle and strength of the cloth remained unaltered. C.O.C.

Control of Slime-forming Organisms
Buckman Laboratories *BP* 809,351

A mixture of an alkali metal or alkaline earth metal cyanodithioimidocarbonate and an alkylene diamine, e.g. an equimolecular mixture of sodium cyanodithioimidocarbonate and ethylenediamine, has excellent inhibitory effect against slime-forming micro organisms at pH 4–8. It is particularly useful in the pulp and paper-making industries. C.O.C.

Emulsions of Water-insoluble Organic Solvents
General Aniline *BP* 809,657

A mixture of (*a*) 50–90 parts by wt. of a water-soluble non-ionic surface active agent containing a polyoxyalkylene chain of < 2 alkenoxy groups and derived from an alkylphenolic compound whose total alkyl C atoms number 4–24, and (*b*) 10–50 parts of the cyclohexylammonium salt of an alkylbenzene sulphonic acid containing at least one Alk of > 2 C, is soluble and stable in water-insoluble organic solvents. It is used to produce "soluble" solvent concentrates which may be readily added to water to produce stable oil-in-water emulsions. The mixture exhibits a synergistic effect as it is superior to either of its components. C.O.C.

New Products developed since November 1957— Alphabetical List of Colouring Materials, Chemicals, and Equipment (IV p. 261)

IV— RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

New Products developed since November 1957— Alphabetical List of Colouring Materials, Chemicals, and Equipment

Amer. Dyestuff Rep., **47**, 819–849 (1 Dec. 1958)

Steric Effects on Mesomerism. XIX— Comparison of some Properties of a Number of *N*-Mono-n-alkyl- and *NN*-di-n-alkyl-anilines

J. A. C. T. Brouwers, S. C. Bijlsma, P. E. Verkade, and B. M. Wepster

Rec. Trav. chim., **77**, 1080–1088 (Nov. 1958)

The ultraviolet absorption spectra, basic strength, mol. refraction, and behaviour on nitrosation and coupling of *N*-mono-ethyl-, -n-butyl-, -n-hexyl-, -n-octyl-, -n-decyl-, and -n-hexadecyl-aniline, and of *NN*-diethyl-, -n-butyl-, -n-hexyl- and -n-octyl-aniline, indicate that no steric inhibition of mesomerism is perceptible. All the above *NN*-di-n-alkylanilines are easily nitrosated under appropriate conditions, an important one of which is that the tertiary amine or its hydrochloride should be readily sol. in the medium used, and as the length of the n-alkyl group increases, this condition grows constantly more difficult of realisation. Sidgwich's statement (*The Organic Chemistry of Nitrogen* (1942), p. 218) that the nitrosation of *NN*-dialkylanilines fails completely if the alkyl groups are large is found to be too general and consequently incorrect. Nitrosation does, however, fail altogether where the presence of a marked inhibition of the mesomeric interaction between the dialkylamino group and the rest of the mol. has to be inferred on the strength of appropriate criteria, as in 2-methyl-*NN*-dimethylaniline, *N*-methyl-*N*-t-butylaniline, and benzoquinuclidine, where there is a strong impediment to the conjugative electron displacements necessary for electrophilic (*para*) substitution. H.H.H.

Quantitative Study of the Arylation of Naphthalene by means of Diazonium Salts and Zinc

B. A. Marshall and W. A. Waters

J.C.S., 381–384 (Jan. 1959)

Free radicals generated by the decomp., with zinc powder, of acetone suspensions of aryldiazonium salts (phenyl, *p*-nitrophenyl, *p*-methoxyphenyl, and *p*-acetamidophenyl) are found to effect homolytic monosubstitution of naphthalene in both the α- and the β-position. The resulting mixtures of α- and β-arylnaphthalenes are separated chromatographically and analysed spectroscopically, and the variation in the $\alpha:\beta$ ratios for the arylations is tabulated and discussed. In accordance with theoretical calculations, which show that naphthalene has a higher electron-availability at α- than at β-positions, heterolytic substitution of naphthalene below 100° always gives much more α- than β-isomer; but whereas the homolytic substitution by the *p*-nitrophenyl group results in an increased percentage of α-cpd., the *p*-methoxy and *p*-acetamido groups, which have opposite electrical character, both definitely depress the $\alpha:\beta$ ratio. H.H.H.

Direct Introduction of the Diazonium Group into Aromatic Nuclei. V— Action of Buffered Nitrous Acid on Aromatic Hydroxy Acids

J. M. Tedder and G. Theaker

J.C.S., 257–262 (Jan. 1959)

Aromatic hydroxy acids are found to react with buffered nitrous acid in two ways, both leading to diazonium salts. The more simple reaction is the direct introduction of the diazonium group as observed (cf. *J.C.S.*, 2573 (1958)) with other phenolic cpd.; but for *o*- or *p*-hydroxy acids the above reaction is wholly or partly superseded by decarboxylation, whereby the diazonium replaces the ejected carboxyl group. Salicylic acid stands between *m*-hydroxybenzoic acid, which undergoes no decarboxylation, and *p*-hydroxybenzoic acid, which yields only the decarboxylated product, as does 4-hydroxy-3-nitrobenzoic acid; it is of interest that the nitro group decreases the rate of decarboxylation only slightly. *p*-Hydroxycinnamic and caffeic acid are likewise decarboxylated, but attempts to prepare ethylenediazonium salts by this means were not successful. H.H.H.

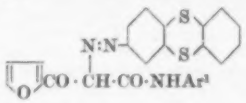
Studies of Furoyl Azo Dyes

VI— Derived from Thianthrene

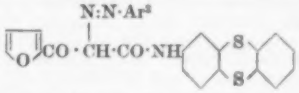
A. Cerniàni

Boll. sci. fac. chim. ind., Bologna, **15**, 69–71 (1957): *Chem. Abs.*, **53**, 1718–1720 (25 Jan. 1959)

Furoylacetarylides couple with diazotised 2-aminothianthrene to give monoazo compounds of the type—



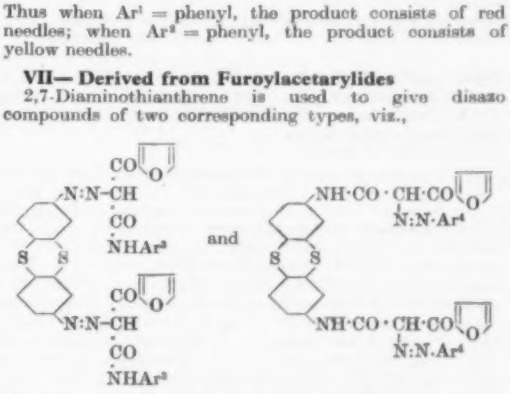
and furoylacet-*N*-2-thianthrylamide couples with diazo compounds to give monoazo compounds of the type—



Thus when Ar^1 = phenyl, the product consists of red needles; when Ar^2 = phenyl, the product consists of yellow needles.

VII— Derived from Furoylacetarylides

2,7-Diaminothianthrene is used to give disazo compounds of two corresponding types, viz.,



the first type giving mainly orange-yellows and the second mainly yellows.

The fastness properties of these dyes on cellulose is good. E.S.

Azo Dye Derivatives of 5-Nitro-1,2-dimethylbenziminazole

M. T. Le Bris and H. Wahl

Compt. rend., **246**, 3472–3474 (1958): *Chem. Abs.*, **53**, 1315 (25 Jan. 1959)

5-Nitro-1,2-dimethylbenzimidazole oxidised with SeO_2 in boiling toluene gives 5-nitro-1-methyl-2-benzimidazolecarboxaldehyde (I). Acylhydrazines treated with I form hydrazones tautomeric with the monoazo derivatives. I condensed with pyridine in presence of iodine and then oxidised with excess nitrosodimethylaniline gives the *p*-dimethylaminophenylnitrone of I. This nitrone can be hydrolysed to yield I. These monoazo derivatives are easily alkylated with alkyl sulphates or iodides to give, after alkalisation, 2-phenylazomethylene-1,3-dimethyl-5-nitrobenzimidazoline and 2-(*p*-nitrophenyl)azomethylene-1,3-dimethyl-5-nitrobenzimidazoline. The alkylated compounds can also be obtained from benzimidazolinium salts through the *p*-dimethylaminoanil of 5-nitro-1,3-dimethyl-2-formylbenzimidazolium methyl sulphate. The *p*-nitrophenylhydrazine of I reacts with diazotised *p*-nitroaniline to give 2-bis(*p*-nitrophenylazo)methylene-1,3-dimethyl-5-nitrobenzimidazoline. 2-Bis(*p*-methoxyphenylazo)methylene-1,3-dimethyl-5-nitrobenzimidazoline was prepared by direct coupling. The contention of Porai-Koshits, *et al.* (*Chem. Abs.*, **50**, 4523 (1956)), that the monoazo derivatives in this series are dealkylated is not established. C.O.C.

Dichroism of Dyes in Stretched Polyvinyl Acetate Film. I— Dichroism of Benzidine Disazo Dyes

Y. Tanizaki and N. Ando

Nippon Kagaku Zasshi, **78**, 542–546 (1957): *Chem. Abs.*, **53**, 3697 (25 Feb. 1959)

Congo Red (C.I. Direct Red 28), Diamine Violet N (C.I. Direct Violet 1), Diamine Black RO (C.I. Direct Black 29), Benzopurpurine 4B (C.I. Direct Red 2), Azo

Blue (C.I. Direct Violet 28), Benzopurpurine 10B (C.I. Direct Red 7) and Benzo Azurine G (C.I. Direct Blue 8) on stretched polyvinyl acetate film have complex spectra with 45° polarised light (angle between vectors of stretched direction and incident polarised light. Change of angles from 0° to 90° caused shift in maximum at shorter wavelength. Dyes from NW or γ-acid (α-coupling) had similar spectra irrespective of the angle, while those from naphthionic acid or γ-acid (β-coupling) changed both in shape and in maximum. The absorption band changed from simple to complex as the medium changed, the clearest example being Benzopurpurine 10B (335 mμ. in water; 320, 345 and 380 mμ. in both ethyl alcohol and poly(vinyl acetate). The band of Benzopurpurine 10B at 325 mμ. increases when the angle is changed from 90° to 0° and that at 350 mμ. decreases. This is caused by presence of two chromophores, each contributing to perpendicular and parallel polarised light. Diamine Violet N has maxima at approx. 300 and 310 mμ. for 90° and 0° respectively and Diamine Black RO has maxima at 300 and approx. 310 mμ. for 0° and 90° respectively. The band at 300 mμ. is caused by the chromophore of short axis direction and that at 310° mμ. by that of long axis. The maxima of longer wavelength were almost constant.

C.O.C.

Dyes derived from Imidazole. I—Preparation and Chromatographic Separation of Dyes from 1,4,5,8-Naphthalenetetracarboxylic and 1,8-Naphthalenedicarboxylic Acids

J. Arient and J. Franc

Chem. Listy, **52**, 1946–1950 (1958): *Chem. Abs.*, **53**, 1722 (25 Jan. 1959)

Condensing 1,8-naphthalenedicarboxylic acid (I) or 1,4,5,8-naphthalenetetracarboxylic acid (II) with the respective diamine by heating for 3 hr., dissolving the products in 100 ml. acetic acid, cooling, diluting with an equal volume of water, and washing the product until neutral yields the following dyes: (component, weight in g., % yield, colour, vatability): I, 10, 3,4-$(NH_2)_2C_6H_3OC_2H_5$ (III) 7·4, 69 yellow, poor; I 10, 1,8-naphthalenediamine (IV) 7·32, 88·5, red, poor; I 10, 1,2-naphthalenediamine hydrochloride (V) 9, 74·6, orange, poor; II —, III —, —, brown, good; II 10, V 12·8, 92·5, violet, poor; II 10, IV 10·64, 84, violet, poor; II 10, V 6·4, 92, —, —, —. The dyes were separated on paper into the individual position isomers. Those with COOH in position 1 or 1 and 4 absorb in longer wavelengths than those with COOH in position 8 or 1 and 5. The first 5 dyes given above were separated on Al_2O_3 and the fluorescence spectra of the isomers determined. Their R_f is approximately in direct proportion to the shift of the maximum and conforms with the relationship between the magnitude of the dipole moments and the R_f value of the respective isomer.

C.O.C.

Pyrrole Dyes. V—Pyrrole Exchange Reactions—Constitution of Dipyrrylmethenes and Porphyrins

A. Treibs, E. Herrmann, E. Meissner, and A. Kuhn

Ann. **602**, 103–183 (1957): *Chem. Abs.*, **53**, 1305 (25 Jan. 1959)

In a study of the constitution of dipyrrylmethenes the addition and substitution reactions of pyrroles, cleavage of substituents at the pyrrole nucleus, formation of symmetrical instead of unsymmetrical substituted dipyrrylmethenes, and their relation to the pyrrole exchange reaction are discussed. Most substitution reactions in the pyrrole exchange reaction are related to the basic properties of the pyrrole nucleus. Salts of pyrrole have the structure of pyrrylene salts. Formation of the latter and their reactivity depend upon the substituents. Substituted dipyrrylmethenes containing negative groups are said to be methene salts, these bases can be isomerised to unsymmetrical dipyrrylethylenes. Pyrrole dyes form both dye cations and dye anions. Synthesis of a large number of pyrrole derivatives and their salts is achieved by condensing pyrrole aldehydes with pyrroles and by the pyrrole exchange reaction. C.O.C.

Scission of the Pyridine Nucleus and its Importance in the Synthesis of Polymethin Dyes

S. Creyf and L. Roosens

Ind. chim. belge, **23**, 853–864, 967–982 (1958): *Chem. Abs.*, **53**, 3699 (25 Feb. 1959)

Review, 213 references. C.O.C.

A Contribution to the Theory of the Optical Sensitisation of Photographic Emulsions. Synthesis of *N*-Phenylbenzimidazoletrimethincyanines

O. Neunhoeffer and A. Keiler

Chem. Ber., **91**, 122–129 (1958): *Chem. Abs.*, **53**, 1965 (10 Feb. 1959)

The excited state of a dye assumes a high positive potential which, in optical sensitisation, induces withdrawal of an electron from a Br ion of the Ag halide and promotes its conduction over the grain surface. This was confirmed by results of a systematic study of a series of *N*-phenylbenzimidazoletrimethincyanines. (Bis[1-(*p*-bromophenyl)-3-ethyl-2-benzimidazole]-trimethincyanine perchlorate sensitises an emulsion more strongly than does the corresponding unbrominated dye, which, in turn, is more effective than the *m*-bromo dye. The tendency of the 1-substituents to withdraw electrons from the positively changed benzimidazole nuclei runs parallel to the sensitisation of the corresponding dyes. Syntheses of a number of these dyes are given. C.O.C.

Condensation Reactions of *meso*Methyl-substituted Trimethincyanine Dyes with Aromatic and Heterocyclic Aldehydes

W. Ried and R. Bender

Chem. Ber., **90**, 2650–2653 (1957): *Chem. Abs.*, **53**, 2245 (10 Feb. 1959)

Luminescence of Dyes in Viscous and Solid Solutions

V. A. Kizel and U. I. Safronova

Optika i Spektroskopiya, **5**, 482–483 (1958): *Chem. Abs.*, **53**, 1918 (10 Feb. 1959)

The relationship between the amount of luminescence and its spectral state was investigated as the function of the viscosity of Fuchsine (C.I. Basic Violet 14), Quinoline Yellow (C.I. Acid Yellow 3), Acridine Yellow (C.I. 46025), Auramine (C.I. Basic Yellow 2), Chrysoidine (C.I. Basic Orange 2), Methyl Orange (C.I. Acid Orange 52), Congo Red (C.I. Direct Red 28), Benzaurin and Pinacyanol dissolved in aqueous glycerol–glucose. In every case luminescence decreases with decrease in viscosity of the solution. This decrease was accompanied by shift of the luminescence band to longer wavelengths, the amount of shift corresponded approximately to the rate in decrease of the amount of luminescence. C.O.C.

Influence of Food Dyes on Lactic Acid Production by Lactic Acid Bacteria

J. Eisenbrand and A. Kauck

Z. Lebensm.-Untersuch. u.-Forsch., **108**, 225–238 (1958): *Chem. Abs.*, **53**, 1573 (25 Jan. 1959)

37 Samples were analysed for actual dye present and titrated with standard base and acid, to serve for correcting for actual dye used and for lactic acid determinations respectively. The results enable the dyes to be placed in 3 classes: (*a*) those with strong bacterial inhibition—Auramine (C.I. Basic Yellow 2), Chrysoidine (C.I. Basic Orange 2), Malachite Green (C.I. Basic Green 4), Methyl Blue (C.I. Acid Blue 93) and Methyl Violet (C.I. Basic Violet 1); (*b*) those giving some inhibition—Eosine (C.I. Acid Red 87), Erythrosine (C.I. Food Red 14), Rose Bengale (C.I. Acid Red 94), Naphthol Yellow (C.I. Food Yellow 1), Orange I (C.I. Acid Orange 20), Ponceau, and Water Blue (C.I. Acid Blue 22); and (*c*) dyes having little or no effect on bacteria (24 listed). In group *b* some dyes showed some inhibiting effect regardless of concentration and with others the effect increased with concentration. Some dyes in group *c* had very slight inhibiting or very slight stimulating effect on the bacteria.

C.O.C.

Physical Properties of Anthraquinone and its Derivatives. I—Infrared Spectra

H. Bloom, L. H. Briggs, and B. Cleverley

J.C.S., 178–185 (Jan. 1959)

The infrared spectra between 2 and 15 μ. of anthraquinone and 59 deriv. of it are reported, and it is found that (1) each spectrum is unique and can be used to "fingerprint" each cpd.; (2) β-hydroxyl groups can be detected by the appearance of a sharp band in the region 3600–3150 cm.$^{-1}$; if there is more than one such band, two or more hydroxyl groups are attached to the anthraquinone nucleus in β-positions, each having a different environment; (3) deriv. with no α-hydroxyl group have one strong carbonyl band in the region 1678–1653 cm.$^{-1}$; (4) deriv. with one α-hydroxyl group have two carbonyl frequencies,

one in the region 1675–1647 cm.$^{-1}$, and an even stronger band between 1637 and 1621 cm.$^{-1}$, the frequency separation between the bands lying in the range 24–38 cm.$^{-1}$; (5) substitution by two α-hydroxyl groups in the 1,4 and 1,5 positions yields spectra with a single carbonyl band between 1645 and 1608 cm.$^{-1}$; (6) deriv. with two α-hydroxyl groups in the 1,8 positions have two bands, one between 1678 and 1661 cm.$^{-1}$ and the other (stronger one) between 1626 and 1616 cm.$^{-1}$, the frequency separation between the peaks lying between 40 and 57 cm.$^{-1}$; (7) substitution by α-hydroxyl groups in the 1,4,5 positions yields a single carbonyl band between 1616 and 1592 cm.$^{-1}$; (8) deriv. with four α-hydroxyl groups, i.e., in the 1,4,5,8 positions, have a single carbonyl band, in the region 1592–1572 cm.$^{-1}$; (9) the average C=C stretching band in anthraquinones of the 1,4- and the 1,5-dihydroxy group has a frequency $>$ 1575 cm.$^{-1}$, while in the case of the 1,4,5-trihydroxy group it is $>$ 1575 cm.$^{-1}$; for anthraquinones of the 1,4,5,8-tetrahydroxy group, the C=O and C=C bands are indistinguishable; (10) it is possible to group the spectra of anthraquinones of the same substitution pattern according to the frequency and intensity of the absorption bands. H.H.H.

Reaction of Amino Derivatives of Anthraquinone, Fluorenone, and Fluorene with Nitrobenzene and α-Nitronaphthalene
W. Bradley and F. P. Williams
J.C.S., 360–363 (Jan. 1959)

Nitrobenzene and α-nitronaphthalene undergo nuclear substitution by arylamino groups *para* to the nitro substituent on being heated with potassium hydroxide and one or other of the amines—1-amino-, 2-amino-, and 2-amino-1-chloro-anthraquinone and 2-amino-fluorenone; the reaction with α-nitronaphthalene occurs more readily than with nitrobenzene. Indanthrone is formed in small amount in the nitrobenzene reaction, but none is found with α-nitronaphthalene, probably because of the greater ease of substitution into the naphthalene deriv. Under similar conditions nitrobenzene and 2-aminofluorene give 2-aminofluorenone and 2-*p*-nitroanilinofluorenone. H.H.H.

Vat Dyes using Acrylonitrile
W. Schmidt-Nickels *J. prakt. Chem.*, **6**, 139–144 (1958): *Chem. Abs.*, **53**, 1721 (25 January 1959)

NN'-Bis(2-cyanoethyl)-bispyrazoleanthrone (red from a blue vat) and *NN'*-bis(2-cyanoethyl)-perylene-3,4,9,10-tetracarboxylic diimide (brown–grey from a violet vat) are prepared by reacting bi-pyrazoleanthrone and perylene-3,4,9,10-tetracarboxylic diimide resp. with CH_2:CHCN and, e.g., aq. tetra-(β-hydroxyethyl)-ammonium hydroxide or aq. benzyltrimethylammonium hydroxide, etc. Preparations of a brominated derivative (15·6% Br) and the leuco-sulphuric ester of the first dye are also given. The dyes are fast to light and chlorine. E.T.

New Pigment (Namakochrome) from the Sea Cucumber, *Polycheira rufescens* (Brandt)
T. Mukai *Mem. Fac. Science, Kyushu University, Japan*, **3**, 29–33 (Oct. 1958)

The mol.wt. (corresponding to $C_{11}H_8O_8$) of the orange–red pigment, its ultraviolet and infrared absorption spectra, and its reactions indicate it to be a new deriv. of the polyhydroxynaphthaquinones, since it is not identical with any of the known naphthaquinone deriv. from sea urchins. H.H.H.

Chemistry of the *Coprosma* Genus. XI—Colouring Matters from *Coprosma foetidissima*
E. G. Brooker *J.C.S.*, 470 (Jan. 1959)

The title substance is a widely occurring small shrub or tree endemic to New Zealand, whose inner aerial bark is bright yellow and contains asperuloside; a light-petroleum extract of it yielded three anthraquinones not yet identified. Extraction with methanol yielded a brown gum, from which rutin was obtained in 9·6% yield; the latter cpd. was also isolated from the root-bark of *Coprosma rhamnoides*. H.H.H.

Chemistry of Carotenoids and of β-Carotene (C.I. 75130)
G. I. Samokhvalov
Tekhnol. i Primenenie Vitamina A i Karotina (S. I. Korol'kova, editor. Gosudarst. Nauch.-Tekh. Izdatel. Minister. Prom. Prodov. Tovarov) 5–9 (1956)

Preparation of Crystalline Carotene and Carotene Oil from Carotene Concentrates
A. S. Vicher *Ibid.*, 15–25

Preparation of Crystalline Carotene by Fractionation of Components of the Protein Coagulant
O. A. Svishchuk and B. G. Savinov *Ibid.*, 25–30

Perfection of the Technology of Carotene Production from Carrots and Gourds
L. O. Shnaĭdman *Ibid.*, 30–39

Certain Peculiarities of the Technology of Carotene Production
B. G. Savinov *Ibid.*, 39–46

Carotenoidal Composition of the Main Vitamin Cultures and Chromatography of Carotenoids
B. G. Savinov *Ibid.*, 46–53

Comparative Evaluation of the Quality of Industrial Preparation of Carotene
Z. S. Grafskaya *Ibid.*, 80–85

Decomposition of Metal-Porphyrin Complexes by γ Irradiation
H. N. Dunning and J. W. Moore
Ind. Eng. Chem., **51**, 161–164 (Feb. 1959)

Chemistry of the Rhodopsin Cycle
T. Yoshizawa and Y. Kitô
Nature, **182**, 1604–1605 (6 Dec. 1958)

Colour and Constitution of Inorganic Solids. I—Light Absorption of Bivalent Cobalt in Oxide Co-ordination Lattices
O. Schmitz-Dumont, K. Brokopf, and K. Burkhardt
Z. anorg. u. allgem. Chem., **295**, 7–35 (1958): *Chem. Abs.*, **53**, 1915 (10 Feb. 1959)

The influence of coordination number, lattice expansion, and changing symmetry of the coordination polyhedron on the light absorption of Co^{++} was studied by analysis of the spectra of the following oxides: $Co_{0\cdot1}Mg_{0\cdot9}Al_2O_4$, $Co_{0\cdot1}Mn_{0\cdot9}Al_2O_4$, $Co_{0\cdot1}Mg_{0\cdot2}Cd_{0\cdot7}Al_2O_4$, $Co_{0\cdot1}Zn_{0\cdot9}Al_2O_4$, $Co_{0\cdot1}TiO_4$, $Co_{0\cdot2}Mg_{1\cdot8}TiO_4$, $Co_{0\cdot1}Mg_{1\cdot9}SnO_4$, $Co_{0\cdot2}Mg_{1\cdot8}SnO_4$, $Co_{0\cdot1}Zn_{1\cdot9}SiO_4$, $Co_{0\cdot25}Mg_{1\cdot75}SiO_4$, $Co_{0\cdot2}Mg_{0\cdot8}TiO_3$, $Co_{0\cdot1}Mg_{0\cdot9}O$, and $Co_{0\cdot0286}Zn_{0\cdot9714}O$. The main absorption bands of Co^{++} are shifted toward longer wavelengths by decrease of coordination number of Co^{++} from 6 to 4 or by increase in the Co–O distance. Decrease in the Co–O distance causes a shift to shorter wavelengths; lattice expansion or contraction does not necessarily change the Co–O distance and thereby the position of the absorption maximum. Lowering of the point symmetry of the coordination polyhedron without simultaneous distortion of the polyhedron causes differentiation and broadening of the main absorption bands but little change in the positions of the maximum. Decreased point symmetry together with distortion of the polyhedron produces sharper, more intense maxima which are shifted toward longer wavelengths. The causes of these phenomena are discussed. C.O.C.

Red Pigments for Coatings
S. Bonnie *Off. Dig. Fed. Paint Varn. Prod. Cl.*, **30**, 1113–1121 (Oct. 1958)

Summary of the chemical structure of red pigments, organic and inorganic, and an indication of their alkali resistance, suitability for baked finishes, and light-fastness (not rated on the Blue Scale). J.W.D.

Aluminium Pigments—Review and Developments
E. Scheller *Off. Dig. Fed. Paint Varn. Prod. Cl.*, **30**, 1088–1095 (Oct. 1958)

The qualities of both leafing and non-leafing types, the refinements in their processing, and the closer quality control now imposed, are discussed. J.W.D.

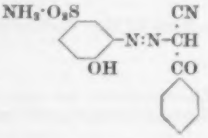
Effect of Particle Size and Shape on the Hue of Ferric Oxide (C.I. 77491)
T. Takada and M. Kiyama
Funtai oyobi Funmatsuyakin, **4**, 187–191 (1958): *Chem. Abs.*, **53**, 3638 (25 Feb. 1959)

An explanation of the variation in hue of ferric oxide based on observations of the reflection and transmission of light with both the dry pigment and in dispersions. C.O.C.

PATENTS

Metal-complex Monoazo Dyes for Wool and Nylon
American Cyanamid Co. *USP* 2,852,502

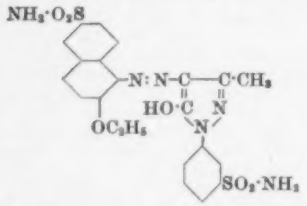
Sulphonamides of *o*-aminophenol and anthranilic acid are diazotised and coupled with aroylacetonitriles and the monoazo compounds converted into 1,2 metal-complexes, which dye wool and nylon from neutral baths. Thus diazotised 2-aminophenol-4-sulphonamide is coupled with benzoylacetonitrile to give



whose cobalt-complex is an orange and whose chromium-complex is a brown. E.S.

Red, Metal-complex Monoazo Pyrazolone Dyes for Wool, Nylon, etc.
General Aniline *USP* 2,853,481

6-Sulphonamides and 6-alkylsulphonyl derivatives of 2-alkoxy-1-naphthylamines are diazotised and coupled with 5-pyrazolones free of SO_3H and COOH groups, and the resulting monoazo compounds metallised with de-alkylation of the 2-alkoxy groups to give Cr, Ni, and Cu complexes which dye wool, nylon, etc. pink or red from neutral dispersions. Thus 2-ethoxy-1-naphthylamine-6-sulphonamide diazotised and coupled with 3-methyl-1-*m*-sulphamylphenyl-5-pyrazolone gives

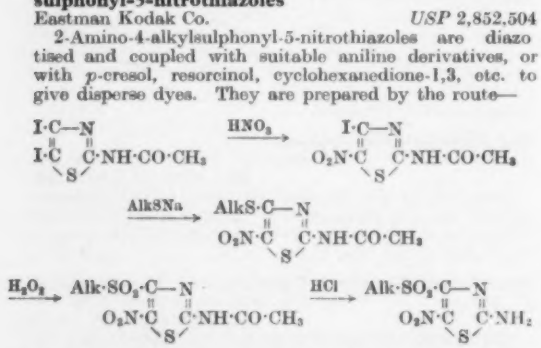


Heating with chromium formate in formamide at 165–170°c. for 5 hr. gives the Cr complex, which after mixing with a dispersing agent dyes wool pink.

Preparations of a number of suitable diazo components are given. E.S.

Monoazo Disperse Dyes from 2-Amino-4-alkylsulphonyl-5-nitrothiazoles
Eastman Kodak Co. *USP* 2,852,504

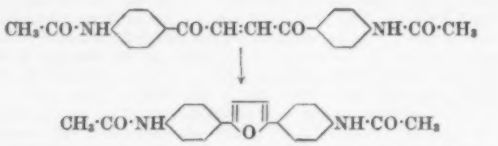
2-Amino-4-alkylsulphonyl-5-nitrothiazoles are diazotised and coupled with suitable aniline derivatives, or with *p*-cresol, resorcinol, cyclohexanedione-1,3, etc. to give disperse dyes. They are prepared by the route—



The shades on cellulose acetate of 69 examples are given, ranging from 2-amino-4-methylsulphonyl-5-nitrothiazole (I)→*p*-cresol (yellow–orange), and I→*NN*-bis-β-hydroxyethyl-*p*-cresidine (blue green). E.S.

2,5-Bis(*p*-Aminoaryl)furans— Fluorescent Brightening Agents and Intermediates for Azo Dyes
American Cyanamid Co. *USP* 2,852,503

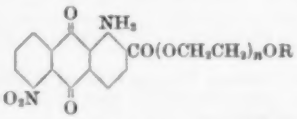
The title products are made by condensing, e.g. acetanilide with fumaryl chloride, followed by ring-closure in a mixture of HBr, acetic acid, and β-naphthol, thus—



the product showing blue fluorescence in solution. Hydrolysis of the acetyl groups gives the diamine, which may be used as a tetrazotisable base for producing azoic dyeings, or sulphonated, tetrazotised, and coupled with suitable coupling components to give direct cotton dyes. E.S.

Red Anthraquinone Disperse Dyes
ICI *BP* 809,043

Bright reds for acetate, triacetate, and Terylene fibres having good light, gas fume and sublimation fastness are



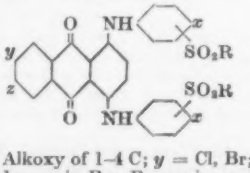
(n = 1–3; R = H, methyl, ethyl or butyl). They are made by (1) heating 5-nitro-1-aminoanthraquinone-2-carboxylic acid with an alcohol, $H(OCH_2CH_2)_nOR$, and, e.g., HCl or H_2SO_4 to promote ester-formation at 80–150°c. or (2) heating 5-nitro-1-aminoanthraquinone-2-carbonyl chloride and the alcohol for a shorter time at lower temperature. E.T.

Halogenated Anthraquinone Compounds
Ciba *BP* 809,222

Compounds which are either vat dyes or vat dye intermediates are made by the action of molecular halogens in anhydrous $AlCl_3$ on alkaline condensation products of Bz-1-halogenobenzanthrones and anthraquinones having α-N atoms carrying replaceable H atoms, e.g. Bz-1-(α-anthraquinonylamino)-benzanthrone or 6-Bz-1-di-(α-anthraquinonylamino)-benzanthrone (see *GP* 517,422). Such starting materials may also have Alk, alkoxy, Hal, amino, acylamino or NO_2 substituents. Reaction occurs in presence of added fluxing agents, e.g. NaCl, SO_2, nitrobenzene, etc., in 30 min. to 3 hr. at 85–135°c. with introduction of 1–6 Hal atoms/mol. of carbonyl compound. The products which are vat dyes have good dyeing properties and good fastness to chlorine. E.T.

Anthraquinone Dyes for Synthetic Polymers
S *BP* 809,491

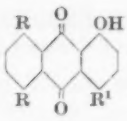
Dyes for the mass coloration of fibres spun from organic solvents, for printing polyester, cellulose triacetate or polyvinyl chloride–acrylonitrile co-polymer fibres or for colouring lacquers and plastics are—



(x = H, Alk or Alkoxy of 1–4 C; y = Cl, Br; z = Cl when y is Cl *or* H when y is Br; R = primary or secondary aliphatic amine of ≮ 3 C attached to the $-SO_2-$ group via the amino N; R may have substituents *or* be the radical of a primary, wholly or partially hydrogenated, mono- or di-nuclear aromatic amine *or* the morpholine radical). The dyes are made by (*a*) reaction at −10 to 100°c. between $ClSO_3H$ and a subst. 1,4-diarylaminoanthraquinone having ≯ 1 SO_3H on the arylamine nuclei to give the corresponding $-SO_2Cl$ compound; (*b*) condensation of the $-SO_2Cl$ compound with an amine at 35–60°c. in presence of an acid-binding agent. E.T.

Gas-Fume Fast Anthraquinone Disperse Blues
General Aniline *USP* 2,850,510

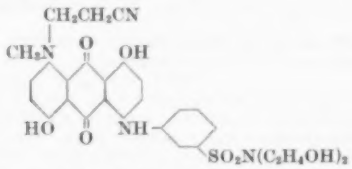
Blue disperse dyes having good affinity and good light and gas-fume fastness on cellulose acetate, polyamide, polyester, and polyacrylonitrile fibres are of formula—



(one R = OH; the other R and R^1 = $-N\begin{matrix}CH_2CH_2X\\R^2\end{matrix}$ *or*

R^1 may = $-NH$; R^2 = lower Alk or $-CH_2CH_2X$;

X = OH, –CN, $–CONH_2$; Y = a substantivity-increasing radical, e.g. hydroxyalkyl, acetamido, sulphonamido, etc.). Preparation is, e.g., by the 2-stage replacement of the nitro-groups of 4,8-dinitroanthrarufin or 4,5-dinitrochrysazin (or a mixture) successively by condensation using (*a*) 2 mol. of a subst. arylamine and (*b*) 2 mol. of a subst. secondary amine. Catalysts, acid-binding agents and inert solvents are used in the condensations. A gas-fast blue so obtained is



10 other examples are given. E.T.

Preparation of Pyranthrone

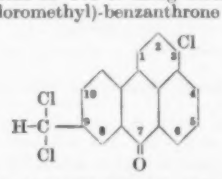
DuP *USP* 2,855,408

Ring closure of 2,2′-dimethyl-1,1′-dianthraquinonyl (1 pt.) to pyranthrone is carried out in presence of NaOH, KOH or K_2CO_3 (≮ 1 pt.), water to give an alkali concentration of ≮ 30% and an organic hydroxy compound (≮ 0·1 pt.) at 110–160°c. An alternative to the organic hydroxy compound is a substance which is converted to such a compound during the reaction, and these hydroxy compounds serve as reducing agents in the system. Examples are β-methoxymethoxyethanol, 2-ethoxyethanol, diethyleneglycol and its mono-methyl ether. Quantitative yields are claimed and products are of higher purity than those obtained by earlier processes, e.g. those of *USP* 856,811 (1907). E.T.

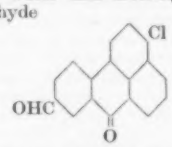
Substituted Benzanthrones

General Aniline *USP* 2,855,409

3-Chloro-9-chloromethylbenzanthrone is chlorinated in ethylene dichloride at 160°c. using chlorine gas to give 3-chloro-9-(dichloromethyl)-benzanthrone



Hydrolysis by heating in 96% H_2SO_4 (2 hr., 70°c.) followed by drowning into water and filtering gives 3-chloro-9-benzanthrone aldehyde



This product is capable of condensing with, e.g., 1-aminoanthraquinone to give a yellow cotton dye of good fastness. E.T.

Sulphur-containing Dyes

BASF *BP* 809,224

Dyes especially suitable for printing and having good light-fastness are

$$D–(CH_2–X–S–R)_n$$

or $D–(CH_2–X–S\overset{Y}{\underset{\oplus R}{}}\ominus Z)_n$

(D = residue of a polynuclear dye containing ≮ 1 CO or C=N group; X = divalent aromatic radical; R, Y = alk; Z = the equiv. of an anion; n = 1–8). Suitable polynuclear dyes are metal-containing bis- to octakis-(chloromethyl)-phthalocyanines or halomethyl-containing indigos, thioindigos, anthraquinones, benzanthrones, pyranthrones, etc. Reaction proceeds thus, e.g.

$$(Dye)–CH_2–Cl + HX–S–R \rightarrow (Dye)–CH_2X–S–R + HCl$$

in presence of catalysts, e.g. $ZnCl_2$, $FeCl_3$, $AlCl_3$, BF_3 at about 100°c. and addition of NaCl or Na_2SO_4 is advantageous. The products may be converted to water-sol. sulphonium compounds. Thus, the thioindigo derivative containing one sulphonium group is water-sol. but Cu phthalocyanine requires two per mol. E.T.

Hydrocarbon-soluble Phthalocyanine Dyes

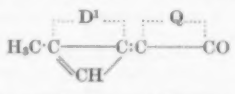
General Aniline *USP* 2,859,219

Treatment of chloro-methylated copper phthalocyanine colorants with alkyl aromatic compounds results in HCl being split off and the alkyl aromatic residue combining with the phthalocyanine to render it soluble in hydrocarbons without impairing its tinctorial power, stability or other valuable characteristics. Thus copper tetrachloromethylphthalocyanine was stirred into a mixture of dodecylbenzene, nitrobenzene and $SnCl_4$ and heated slowly under reflux to 115°c. and the temperature maintained at 115–125°c. for 4 hr. The mixture was cooled, diluted with a large excess of ethanol, filtered, and the precipitate washed with ethanol and then with water and finally oven dried at 80–90°c. The dye was purified by dissolving in warm benzene, filtering, diluting with ethanol, washing the precipitate with ethanol and drying. The product is valuable as a colorant for petrol, printing inks, and hydrophobic resinous polymers, especially those of the polyvinyl series, e.g. polystyrene. It has excellent fastness to light. C.O.C.

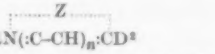
Merocyanine Dyes

Eastman Kodak Co. *USP* 2,856,404

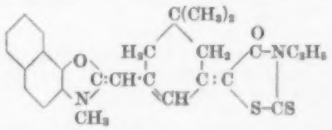
Condensation of a compound of formula—



(D^1 = atoms to complete a cyclohexene ring; Q = atoms to complete an indandione or 5- or 6-membered heterocyclic ring) with one of formula—



(R = Alk; n = 0 or 1; X = acid radical; D^2 = electronegative radical) yields a merocyanine dye. Thus the dye



is obtained by condensing the quaternary salt of 2-methylmercapto-α-naphthoxazole and methyl *p*-toluenesulphonate with 3-ethyl-5-(3,5,5-trimethyl-2-cyclohexen-1-ylidene)rhodanine in pyridine with triethylamine present. C.O.C.

Quick Reduction of Pigments to Very Fine Particle Size

DuP *USP* 2,857,400

The dry crude pigment is given a short treatment in grinding apparatus having both attriting and shearing action, e.g. a ball mill, and is then violently agitated in an inert, non-corrosive organic liquid, preferably by passing a slurry of the dry-ground pigment in acetone through a homogeniser or colloid mill. This greatly reduces the time needed to reduce the pigment to the required fine particle size. C.O.C.

Continuous Production of Carbon Black (C.I. Pigment Black 6 and 7)

Godfrey L. Cabot *BP* 809,802

Gaseous hydrocarbon, preferably hot, is introduced into an unobstructed, heat-insulated reaction zone into which

hydrogen heated to above the dissociation temperature of the hydrocarbon is introduced. Dissociation of the hydrocarbon is effected by heat transfer from the hot hydrogen to produce Carbon Black and hydrogen. The resultant gases are cooled and the pigment collected. Some of the gas is reheated and returned to the reactor while the remainder is recovered for other use. C.O.C.

Caramel (C.I. Natural Brown 10)
Union Starch & Refining Co. *BP* 809,747

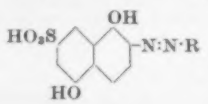
If maize sugar is first modified by mild treatment with hot alkali followed by neutralisation with acid, caramelisation can be carried out to a high degree of colour intensity without encountering the difficulties of thickening and extremely high viscosity. The product is readily diluted with water. C.O.C.

Non-leafing Aluminium Flake Paste (C.I. Pigment Metal 1)
Metals Disintegrating Co. *USP* 2,858,230

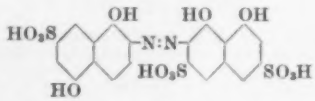
Aluminium is reduced to flake in presence of a lubricant, e.g. a grease, while it is being treated with an aqueous solution of phosphoric acid or an ammonium, alkali metal, alkaline earth metal, zinc or magnesium phosphate. The solution should be at pH 1·5–11·0 and the amount of available phosphate ion should be $\leqslant 0{\cdot}7\%$ by weight of the aluminium. The product is a stable aqueous paste. C.O.C.

Grey Copperable Trisazo Direct Dyes
S *BP* 809,279

Tetrazotised dianisidine is coupled with 1 mol. of a monoazo compound (which may be coppered)—



(R = radical of a 1,8-dihydroxynaphthalene-mono- or -disulphonic acid) and with 1 mol. of the same or other *o*-coupling hydroxy compound, to give direct greys which may be coppered in substance or on the fibre, similar to those of *BP* 695,534 (J.S.D.C., **69**, 385 (1953)) in which R = a radical of a naphtholsulphonic acid. Thus tetrazotised dianisidine is coupled first with 1 mol. of R acid, and then, in presence of pyridine, with 1 mol. of the monoazo compound—



The trisazo dye so formed is heated with ammoniacal $CuSO_4$ in presence of diethanolamine to give the copper-complex which dyes cotton blue-grey. E.S.

Carbon Black (C.I. Pigment Black 6 and 7)
Phillips Petroleum Co. *BP* 809,538

Improvement on the type of processes described in *BP* 621,031 and 679,818 (J.S.D.C., **65**, 418 (1949); **68**, 528 (1952)) whereby the pigment is produced with increased facility and improved heat economy. C.O.C.

Dispersing Pigments in Liquid and/or Reducing Their Particle Size (V this page)

Intramolecular Hydrogen Bonding in Heterocyclic Systems (VIII p. 268)

Decomposition of *o*-Aminoazobenzene by Ultraviolet Radiation (VIII p. 268)

Direct Dyeing of Cellulose. V— Solubility of Direct Dyes in Water (VIII p. 268)

Azoic Dyeings on Polyester Fibres (VIII p. 270)

Spectrophotometric Investigation of Solutions of Dyes forming *H*-Aggregates (IX p. 270)

Sensitisation of Mercurous Oxalate by Dyes (IX p. 271)

V— PAINTS; ENAMELS; INKS

Effect of Extenders on the Hiding Power of Titanium Pigments
F. B. Stieg *Off. Dig. Fed. Paint Varn. Prod. Cl.*, **31**, 52–64 (Jan. 1959)

Assuming a uniform distribution of spherical particles, experimental hiding-power data agree well with the theory that the hiding power of individual particles of white titania is a function of their distance of mutual separation. The hiding power produced per lb. of such a pigment may be calculated from simple formulae based upon this spacing. Fine-particle-size extenders increase hiding by separating the titania particles in a paint film, while large-particle-size extenders tend to reduce hiding power by crowding them together. Calcium-base titania will yield the same result as fine-particle-size extenders, and with greater economy. J.W.D.

Weathering Properties of White Lead and Zinc Oxide Paints
J. R. Rischbieth and K. R. Bussell
J. Oil & Col. Chem. Assocn., **41**, 862–868 (Dec. 1958)

Mechanism of Ink-splitting during Printing
J. H. Taylor and A. C. Zettlemoyer
Tappi, **41**, 749–757 (Dec. 1958)

The quantity of ink transferred from the plate to the paper consists of two portions— one immobilised while the ink is under pressure and a portion of the free ink film which splits towards the stock as stock and plate separate, the split being closer to the stock than to the plate. When a cylinder rolls over a plane surface coated with a fluid film, pressure gradients are established within the film causing local regions of high shear rate adjacent to regions of low shear rate within the film. In a thixotropic fluid this leads to zones of high and low viscosity within the film. When plate and cylinder begin to separate, tension is created within the film and cavities are formed. The point of greatest tension is nearly halfway between the surfaces and cavity initiation is probably greatest in the centre of the film. Cavity growth and ultimate film split proceeds most rapidly in the direction of reduced viscosity, towards the stock. An attempt is made at a mathematical evaluation. 12 references. R.A.

PATENTS

Dispersing Pigments in Liquid and/or Reducing Their Particle Size
DuP *BP* 810,005

A slurry of film-forming material and pigments is fed upwards through a rotated and agitated bed of sand of 20–40 U.S. mesh size. The process is continuous and provides a simple, cheap and efficient method of producing very stable dispersions and/or grinding pigments. C.O.C.

Non-caking Flatting Agent for Varnishes
W. R. Grace & Co. *USP* 2,858,284

A fully shrunk silica gel or silica xerogel which on settling in the varnish forms a soft and flocculent cake which is readily dispersed again throughout the varnish is obtained by forming a silica gel in the conventional manner, washing it free of soluble salts and then rapidly drying it in an oven or a stream of hot gas without regard to preserving the original gel structure. The product, which contains 20% water, is ground to an average of 1–15 μ. with limits of 1–50 μ. Before incorporation in the varnish it is impregnated with sufficient of a low mol.wt. alcohol, e.g. anhydrous methyl or ethyl alcohol, to form a gel containing 50% alcohol. C.O.C.

Pigment Vehicle for Coating Glass
General Electric Co. *USP* 2,858,234

An alkyl acetate together with a small amount of cellulose nitrate and an alkyl lactate is a good vehicle for pigments to be applied to glass, there being little or no tendency for streakiness to develop in the coatings obtained by its use. C.O.C.

Luminescence of Dyes in Viscous and Solid Solutions (IV p. 262)

Red Pigments for Coatings (IV p. 263)

Effect of Pigment Volume Concentration on Coating Properties (XI p. 272)

VI— FIBRES; YARNS; FABRICS

Textiles— Progress in 1958
A. E. Brown and J. F. Krasny
Ind. Eng. Chem., **51**, 57 A–59 A (Jan. 1959)

Chromatographic Analysis of Fractions from Primary Flax Cells
A. Parisot and J. Derminot
Bull. Inst. Text. France, (77), 7–25 (Oct. 1958)

Primary flax cells, both undegraded and after subjection to repeated cycles of hypochlorite treatment, were fractionated into insoluble (I), soluble–precipitatable (II), and permanently soluble (III) material by the action of 10 N.-NaOH gradually diluted to 2 N. and subsequent neutralisation of the solution with acetic acid. Fractions I, II, and III were hydrolysed in 80% formic acid, and the hydrolysates chromatographed on paper in phenol–water. Xylose and arabinose were present in all three fractions from both undegraded and hypochlorite-degraded cells. Fractions II and III contain a higher proportion of xylose and arabinose than does I. Xylose and arabinose appear to exist as such in the cellulose chain, and are tentatively identified with the reactive sites in the chain. J.C.F.

Australian Research on Wool. I— Structure and Chemistry of Wool
F. G. Lennox *Research*, **12**, 82–90 (March 1958)

A comprehensive review of the work carried out during recent years in the Melbourne laboratories of the Division of Protein Chemistry (formerly Biochemistry Unit), C.S.I.R.O., on the structure and chemistry of wool. 93 references. C.O.C.

Diazo Coupling Reaction of Wool
I. Shimizu, N. Koyama, and I. Sakaguchi
Shinshu Daigaku Sen Igakubu Kenkyû Hôkoku, **6**, 126–130 (1956): *Chem. Abs.*, **53**, 1727 (25 Jan. 1959)

Wool couples best with diazotised aniline in 0·30–0·32 N-NaOH, time of treatment being 120–150 min. The increase in weight suggests that coupling occurs with the tyrosyl, histidyl and some other groups in the wool substance. Chemically-treated wools, e.g. methylated, diaminised, chlorinated, and acid-treated, readily couple with diazotised aniline in weaker solutions of NaOH. The regain of azo wool is less than that of the original wool. When the azo wool was kept dry for a week, it broke into smaller and smaller pieces until their length equalled the diameter of the fibre. C.O.C.

Decomposition of Silk Fibroin by Sunlight. IV— Effect of Light on Tyrosine and Tryptophan
S. Okamoto and T. Yamaya
J. Soc. Textile Cellulose Ind. Japan, **13**, 683–686 (Oct. 1957)

To confirm that yellowing of silk fibres by light is based upon the photochemical changes of tyrosine groups in fibroin, the authors prepared a tyrosine polymer, applied it to white rayon cloth, and then irradiated it with an Atlas Fade-O-meter. Yellowing of the cloth was hardly visible. Colour reactions such as Neubauer–Rhode's, Nessler's, or xanthoprotein reactions were looked for on the treated cloth but they gave results very different from those of silk fabrics. On adding a small quantity of tryptophan, however, the states of yellowing and other reactions became similar to those of silk fabrics. The yellowing of proteinaceous fibres is therefore not due to changes in the tyrosine but to tryptophan. The tyrosine residues of the polymer were, however, rapidly decomposed by light. The decomposed products contained some unknown substances coloured by Pauly's diazo reagent, but neither amino, imino acids, indole compounds, nor other expected substances were identified. L.P.

Reducing the Light-tendering of Nylon with Phenols
T. Hashimoto *J. Soc. Textile Cellulose Ind. Japan*, **14**, 321–325 (May 1958)

After 40 hr. exposure in a Fade-O-meter, nylon taffeta suffered a 40% loss in tensile strength. If treated below 60°c. with an aqueous solution of phenol (0·5%) the loss was only 23%. Solutions of *m*-cresol, 2,4,6-trichlorophenol, Na pentachlorophenoxide, *o*-hydroxyacetophenone or 2,4,5-trihydroxyacetophenone act similarly. With α-naphthol the loss was 55%, but with β-naphthol it was 30%, while gallic acid made it 10%. Catechol, resorcinol, or quinol reduced the loss to 1, 4, or 3% respectively. Phenol treatment increased the warp shrinkage of unset taffeta from 7 to 10·5% as the concn. increased from 0 to 2%, whereas with unset nylon it was increased from 0·5 to 4%. After extraction for 1 hr. at 80°c. the unset material suffered warp shrinkages increasing with concn. from 7·5 initially to 15% in 2% phenol, while in the set material they changed from 1·5 to 8%. The tensile strength of the nylon is not affected appreciably by phenol treatment for 1 hr. at room temp. until the concn. exceeds 2%, but the elongation at break increases from 48% initially to 58% at 2% concn., and is 90% at 4% concn. of phenol. The partition coefficient of phenol between nylon and water is 11·0 at 40°c., 9·6 at 60°c., and 7·6 at 80°c. The absorption rate constants for 3-denier drawn yarn are 0·012, 0·024, and 0·060 mg./g. nylon per min. (at the above temperatures) and for 60-denier undrawn yarn they are 0·007, 0·016, and 0·030 respectively. Heat treatment at 190°c. for 30 sec. causes a tenfold reduction in the rate constant at 40°c., and 2 min. heating lowers it further. L.P.

Reducing the Light-tendering of Nylon with Copper
T. Hashimoto *J. Soc. Textile Cellulose Ind. Japan*, **14**, 325–328 (May 1958)

Untreated nylon absorbs Cu according to a Langmuir adsorption isotherm, but ultraviolet irradiation or acetylation reduces take-up from 13 to 0·1 mg. Cu/g. nylon. The rate of absorption of Cu by nylon from $CuSO_4$ soln. is slow at 40°c. (0·6 mg./g. after 20 hr.), but is more rapid at 60° and 80°c., reaching 1·0 mg./g. after 1 hr. Tendering after 40 hr. exposure in a Fade-O-meter is reduced from a 40% loss of tensile strength to ~ 10% by treatment with 1% $CuSO_4$ (on wt. of fibre) for 2 hr. at 60°c. or for 1 hr. at 80°c. Soaping (0·5% soap + 0·3% soda soln.) at 80°c. for 30 min. removes only a small amount of Cu and leaves the light-tendering virtually unchanged at < 10%. Acetic acid (1% on wt.) reduces the amount of Cu absorbed from 1·5 to ~ 0·1 mg./g., but the strength loss after exposure is still only about 7%. Cu absorption increases to 1·6 mg./g. in 0·3% NH_3 and to 3·5 mg/g. in 30% NH_3 (on wt.) with but little effect on light-tendering (5·5% loss in 3% NH_3, 2·9 mg. Cu/g. nylon). L.P.

Effect of Thermal Treatments on the Dyeability of Thermoplastic Yarns (VIII p. 269)
Influence of the Properties of Polyacrylonitrile Fibres on the Conditions of their Dyeing (VIII p. 269)

VII— DESIZING; SCOURING; CARBONISING; BLEACHING

Effect of Sodium Sulphite on Capillary Properties of Cotton Fabrics in Kier Boiling
V. M. Bel'tsov *Izvest. Vysshikh Ucheb. Zavedenii, Tekhnol. Tekstil. Prom.*, (1), 153–156 (1958): *Chem. Abs.*, **53**, 1725 (25 Jan. 1959)

Addition of 20 g./l. Na_2SO_3 to the kier liquor did not increase the capillary properties and stability to heat of cotton fabrics. A more than normal amount of Na_2SO_3 did not render cotton more stable to oxidation. 20 g./l. Na_2SO_3 decolorises the kier liquor and yields goods 2–3% whiter. Na_2SO_3 should normally be used only for excessively dirty goods. C.O.C.

Aspects of Hypochlorite Bleaching
J. C. Paulson *Tappi*, **41**, 655–657 (Nov. 1958)

The progress of the two main reactions in the first stage hypochlorite treatment of prehydrolysed sulphate pulp, i.e. delignification and carbohydrate depolymerisation, were followed by determining the time rate of hypochlorite consumption and the change in pulp fluidity versus the amount of hypochlorite consumed. The period of initial rapid hypochlorite consumption is due to a reaction involving lignin, while the period of slower hypochlorite consumption is accompanied by attack on the carbohydrate fraction. The reaction is temperature-dependent. R.A.

Bleaching the Hair of Marmot Skins
K. A. Barmina *Nauch.-Issledovatel. Trudy Nauch.-Issledovatel. Inst. Mekhovoĭ Prom.*, **6**, 161–173 (1955): *Referat. Zhur., Khim.* (1956), Abstr. No. 56792: *Chem. Abs.*, **53**, 3702 (25 Feb. 1959)

The skins are given a formalin–brine tannage followed by treatment with hyposulphite. This renders the leather resistant to oxidising–reducing treatments. They are

then mordanted with 6 g./l. of Fe_2SO_4, treated with hydrosulphite and bleached with 30 ml. 30% H_2O_2/l. in presence of 8 ml./l. NH_4OH. The pH of the bleaching liquor should be 7·5–7·8 at the start and 6·3–6·7 at the conclusion. The bleaching liquor should be at 28–30°C. Dark brown hair takes 3 hr. to bleach and black 5 hr. C.O.C.

PATENTS

Continuous Bleaching of Cotton Cloth with Hydrogen Peroxide
DuP *USP* 2,858,183

The cloth is treated with an aqueous solution of a phosphate and a wetting agent at 120–140°F., rinsed and then treated with alkaline peroxide solution, steamed to bring to 205–212°F. and then stored in an insulated J-box for 30–90 min.; finally it is rinsed. C.O.C.

Bleaching Cellulose with the Vapours of Hydrogen Peroxide and Water
DuP *USP* 2,859,087

A moving web of fibrous cellulose is separately and continuously treated with (*a*) a liquid containing a non-volatile water-soluble conditioning agent and/or a stabiliser, e.g. a mixture of caustic soda and sodium pyrophosphate, and (*b*) hydrogen peroxide vapour, and then subjecting the web to conditions under which bleaching will occur. The method is particularly applicable for integration with the usual process of paper making. C.O.C.

Bleaching of Coir
Amsterdamsche Maatschappij tot Exploitatie van Octrooien *USP* 2,857,238

Coir which has been treated with sodium hydrosulphite immediately after it has been freed from the surrounding husk is a nearly 100% white durable material. When it is further bleached with a chlorite a pure white fibre is obtained. C.O.C.

Wool Washing
Stiftelsen Svensk Textilforskning *BP* 810,134

Wool is impregnated with washing liquid and is then fed onto a vibrating perforated bed beneath nozzles which spray further washing liquid onto it. The vibrations of the bed cause the wool to move away from the end on which it is fed and off at the other end. The washing liquid is continuously circulated from a container beneath the bed to wool ready to be fed onto the bed, a portion of the liquor is however drawn away from the main circuit, pumped and fed to the nozzles. The process is very suitable for use of organic liquids, e.g. the mineral oils of *BP* 668,467 (J.S.D.C., **68**, 227 (1952)), as the washing liquid. C.O.C.

Bleached Worsted Tops—Chemical Investigation (XIV p. 275)

VIII— DYEING

Intramolecular Hydrogen Bonding in Heterocyclic Systems
E. W. Gill and E. D. Morgan
Nature, **183**, 248–249 (24 Jan. 1959)

The theory is put forward that intramolecular hydrogen bonding has a much higher covalent character in heterocyclic systems where there are extended double bond systems capable of giving resonance than in intermolecular bonding, where this possibility is excluded. The great shift in infrared absorption band in appropriate cases and intramolecular distances are quoted in support of this argument. W.J.M.

Dyeing by Forming a Molecular Compound or Condensation Product in the Fibre
K. Yamada and C. Shinomiya
J. Soc. Textile Cellulose Ind. Japan, **14**, 183–187 (March 1958)

A mixture of substances which can form a molecular compound or condensation product, such as trinitrobenzene–α-naphthylamine or chloranil–benzidine is heated with the fibre in water. With molecular compound formation, dyeing of synthetic fibres such as vinylon and vinyl chloride sheet was possible, though the light fastness was not high. Condensation dyeing, however, showed more promise with both natural and synthetic fibres, and among 5 quinones and 14 amines, *p*-quinones were better than their *ortho* isomers, chloranil was inferior to *p*-benzoquinones for cellulose, while aromatic primary diamines, having conjugated double bonds, were better than the non-conjugated. Aliphatic amines, secondary and tertiary amines are inferior to aromatic primary ones. The presence of acid groups increases water solubility and levelling power, but usually reduces affinities. The range of hue in these series was found to be from 570 to 620 mμ. in the dominant wavelength. L.P.

Effect of Dye, Fibre, and Atmosphere on Light Fastness
G. Schwen and G. Schmidt
J.S.D.C., **75**, 101–105 (Feb. 1959)

Decomposition of *o*-Aminoazobenzene by Ultraviolet Radiation
I. Rusznák, M. Fehérvári, L. Tölgyesi, and G. Bán
Magyar Textiltech., (2), 73–74 (1957): *Hungarian Tech. Abs.*, **10**, (2), 38 (1958)

o-Aminoazobenzene is decomposed by ultraviolet radiation with formation of phenol and *o*-quinone, which are also the final products of its chemical oxidation. Comparison of the fading of *p*-aminoazobenzene and *o*-aminoazobenzene indicates that decomposition is less extensive and much slower with the latter isomer. The experiments confirm the assumption that the photodecomposition of *o*-aminoazobenzene—like that of *p*-aminoazobenzene[2]—is an oxidation process, and they prove that light fastness is increased by the steric effect of the *ortho* substituent. The decomposition of the dye starts at the azo group, and an amino group in the *ortho* position to the former *quasi* protects the azo group from oxidation. It may be assumed that hydrogen bonding takes place between the nitrogen atoms of the azo and amino groups, and a chelate structure is formed. C.J.W.H.

Fast Solid Dyeings on Cotton
M. R. Fox *Dyer*, **121**, 101–108 (16 Jan. 1959)

The general problem of obtaining solid dyeings on material containing neps is discussed and the behaviour of the different classes of dyes is described. The problem fundamentally is that the immature and exposed cotton neps are partially stripped during rinsing. Thus the more rapidly diffusing dyes give light neps. A test based on this observation can be used to indicate immature cotton neps using rapidly and slowly diffusing dyes, e.g. Durazol Red 2B (ICI) and Durazol Green D (ICI), and washing off under controlled conditions. While direct cotton dyes follow this logical scheme, vat dyes show no systematic relationship with dyeing properties, and these dyes are classified separately according to their suitability in practical tests. A number of examples of the effect of dyeing method on the coverage of dead cotton and abrasion coverage are given, and it is found that padding methods are superior. Sulphur dyes in general cover this type of fabric fault well, while azoic combinations are poor. The effect of cloth prepare is also discussed, and particular attention is drawn to the effect of tension. Dye absorption can vary by as much as 2·63 times, comparing dyeings carried out on material prepared and dyed under tension and material prepared and dyed under relaxed conditions. The extent of the variation depends on the levelling power of the dye. Mercerising before dyeing will frequently level up certain types of fibre irregularities. W.J.M.

Direct Dyeing of Cellulose. V— Solubility of Direct Dyes in Water
K. Nishida *Sen-i Gakkaisha*, **14**, 865–868 (1958): *Chem. Abs.*, **53**, 1725 (25 Jan. 1959)

The order of decreasing solubility of some direct dyes is: Congo Red (C.I. Direct Red 28), Nippon Sky Blue (C.I. Direct Blue 15), Aizen Direct Blue BBH (C.I. Direct Blue 6), Diamine Blue 3B (C.I. Direct Blue 14), Dianil Azurine G (C.I. Direct Blue 8), Benzopurpurine 4BKX (C.I. Direct Red 2), Azo Blue (C.I. Direct Violet 39), Azoorseillin. The affinity of these dyes for cotton first increases with decrease in solubility and then decreases. The abnormally low solubility of Azoorseillin and Azo Blue is caused by their chelate structure. C.O.C.

Desorption of Dye from Cellulosic Fibres
Y. Watanabe and K. Miyasaka
J. Soc. Textile Cellulose Ind. Japan, **13**, 765–772 (Nov. 1957)

The amount of colour stripped from a fibre dyed with a selected direct dye varies with the state of the dye on the

fibre. Desorption of dye is a complex phenomenon. Of the many factors, the microstructure of the fibre, the type of dye present, and the conditions of dyeing seem to be the most important. Colour stripping is large at first but decreases rapidly with further wet treatments. A further marked colour stripping is observed when the test-pieces are subsequently treated with a hot alkali solution. It is thought that part of the dyes are fixed mechanically and others physicochemically or chemically, the part fixed mechanically affecting mainly the colour stripped by the hot water treatment, and that fixed physicochemically greatly influencing the colour stripped by the hot alkali treatment. Fibres dyed in weak alkaline dyebaths containing Glauber's salt show colour stripping much more than those dyed in aqueous dyebaths. This is thought to be due to the increase of dye which is fixed mechanically by the coagulation effects of the Glauber's salt. Accordingly, the rate of exhaustion of a dyebath should be kept as low as possible in the early stages of dyeing so that gradual and even distribution of dye can occur in the microstructures of the fibres, and the gradual addition of electrolytes to the dyebath at the final stages of dyeing is the best dyeing procedure. Dyeing of the crystalline regions was found to give different desorption phenomena. L.P.

Flash Photolysis of Vat Dyes
N. K. Bridge and W. M. Maclean
J.S.D.C., **75**, 147–153 (March 1959)

Vat Dyeing of Wool and Nylon
N. G. Klemin and P. V. Moryganova
Izvest. Vyshikh. Ucheb. Zavedenii, Tekhnol. Tekstil. Prom., (1), 142–152 (1958): *Chem. Abs.*, **53**, 1724 (25 Jan. 1959)

Potentiometric titrations of the leuco dye before and after equilibrium dyeing of nylon with the vat dye, Brilliant Violet K, show that the interaction between dye and fibre at pH 11·2 can be represented as—

$$\overset{+}{H}\cdot\bar{O}OC\text{-}K\text{-}\overset{+}{N}H_3\cdot\bar{O}H + \bar{O}\overset{\diagup\!\!\diagup}{C} \rightleftarrows \overset{+}{H}\cdot\bar{O}OC\text{-}K\text{-}\overset{+}{N}H_3\cdot\bar{O}\overset{\diagup\!\!\diagup}{C} + \bar{O}H$$

(K = nylon). Vat dyeing of wool in presence of triethanolamine yielded brilliant dyeings of very good fastness and little damage to the wool. The good fastness is caused by chemical interaction between the leuco dyes and keratin. Not only were stable leuco compounds formed with $Na_2S_2O_4$ or Na formaldehydesulphoxylate but many polycycloketone vat dyes were reduced in presence of triethanolamine. Nine diagrams indicate the relationship between the amount of vat dye on the fibre and the various factors which are of importance in dyeing. C.O.C.

Dyeing of Wool at Room Temperature
B. S. Harrap J.S.D.C., **75**, 106–107 (Feb. 1959)

Durable Pleating of Wool Fabrics— Effect on Dyes
G. M. Swinton J.S.D.C., **75**, 153–154 (March 1959)

Photochemical Degradation of Black-dyed Silk
P. Rochas and Mme. Pierret
Bull. Inst. Text. France, (77), 99–109 (Oct. 1958)

Measurements of fluidity and dynamometric resistance show that black dyeing protects silk against photochemical degradation, provided that chemical degradation has not been caused during the dyeing operation. J.C.F.

Heat Fastness of Dyes on Synthetic Fibres
A. Würz *Z. ges. Textilind.*, **60**, 1017–1024 (Dec. 1958)

Dyes and dye components of low molecular weight exhibit appreciable sublimation under the conditions of heat-treatment of synthetic fibres. Such heat-treatment may be subdivided according as the heating is by contact, hot air, or steam. A small laboratory press for the study of contact heat fastness is described, and data are given for the following ranges of dyes— Palanil, Ortolan, Vialon, Perliton, Celliton, and Basacryl. A short section on hot air fastness includes data for Palanil dyes. Fastness to steam will be treated in a later article. Fastness assessments are based on the grey scale of the Deutsche Echtheitskommission. J.C.F.

Mechanism of the Dyeing of Polyester Fibres with Disperse Dyes
O. Glenz, W. Beckmann, and W. Wunder
J.S.D.C., **75**, 141–147 (March 1959)

Effect of Thermal Treatments on the Dyeability of Thermoplastic Yarns
AATCC
Amer. Dyestuff Rep., **47**, P 853–P 862 (1 Dec. 1958)

Experiments carried out on acrylic, polyamide, polyester and cellulose triacetate fibres show that "dyeability" decreases with increasing temperature of treatment. Subsidiary experiments on triacetate and polyamide yarn show that, while tension has little effect, steam treatment increases considerably the severity of the effect. From X-ray data, density measurements, and viscosity measurements it is concluded that the changes are due to physical differences in orientation and crystallinity rather than to chemical degradation. It is also noted that the sensitivity to temperature variations during heat-treatment is affected greatly by the temperature of the treatment, and if the conditions are suitably selected quite wide limits can be tolerated. On the other hand, the accuracy required within certain temperature ranges to avoid variable dyeing properties is greater than is normally attained under works conditions. W.J.M.

Influence of the Properties of Polyacrylonitrile Fibres on the Conditions of their Dyeing
S. P. Mateeva, B. E. Geller and A. B. Pakshver
Nauch. Dokladŭ Vysshei Shkoly, Khim. i Khim. Tekhnol., (3), 553–555 (1958): *Chem. Abs.*, **53**, 1723 (25 Jan. 1959)

The ability of a fibre to absorb dye at different rates, depending on the degree of swelling, can be used in practice for estimating the uniformity of the fibre structure. When a fibre is dyed in a 0·01% dye liquor at 85–100°c. and liquor ratio 10 : 1 for 30–60 min. the less the fibre has been stretched during its manufacture the deeper the dyeing; direct dyes show the greatest difference. The capacity of a fibre to be dyed by different classes of dyes while drying can be used for the dyeing of polyacrylonitrile fibres during their formation. The freshly-formed fibre after stretching and washing, but before drying, is dyed deeply by direct, acid, basic, and disperse dyes. After it is dried the capacity of the fibre to absorb dye from a dyebath has practically disappeared. To obtain a deep dyeing the amount of dye should be 3–5% on the weight of the fibres. Increase in the size of the diffusing particles of dye and decrease in the swelling of the fibre and its included water, result in decreased rate of absorption. The fastness of the dyeing depends on the temperature of drying. Fibre dried at 22°c. when treated with 0·5% soap solution lost 22% by weight of its absorbed dye, dried at 80°c. 14%, and at 120°c. only 8·4°. Freshly-formed and fresh-washed polyacrylonitrile fibres, kept for 14 days in rubberised bags, did not lose their capacity for being dyed by direct and acid dyes. C.O.C.

Colouring Fatty Foods with β-Carotene (C.I. Natural Yellow 26)
J. C. Bauernfeind, E. G. Smith, and R. H. Bunnell
Food Technol., **12**, 527–535 (1958): *Chem. Abs.*, **53**, 2498 (10 Feb. 1959)

Description of the properties and uses of six forms of special β-carotene products. The effectiveness of β-carotene as a colorant for butter, margarine, shortening, baked products, egg yolk, Cheddar and process cheese is shown. The fine crystalline forms of β-carotene rapidly dissolve in the fats at 100–130°F. C.O.C.

Colouring Aqueous Foods with β-Carotene (C.I. Natural Yellow 26)
R. H. Bunnell, W. Driscoll, and J. C. Bauernfeind
Food Technol., **12**, 536–541 (1958): *Chem. Abs.*, **53**, 2498 (10 Feb. 1959)

Stable, dry β-carotene was prepared by emulsifying a supersaturated solution of β-carotene in vegetable oil with a solution of a gellable colloid and drying the emulsion into fine beads. The resultant product disperses readily in aqueous systems and has good stability when stored. Kept at 113°F. for 6 weeks in an open container, the product retains 95–100% of β-carotene. After 6 months at 75°F. 87–100% was present and after 12 months 82–90%. This form of β-carotene was successfully used to colour citrus beverages, Cheddar cheese in the vat, frozen and dried egg yolk, egg nog, ice cream, and dry cake mix. C.O.C.

PATENTS

Treating Nylon with Amines to increase its Affinity for Acid Dyes

DuP *USP* 2,855,267

Nylon that has been treated with compounds of formula

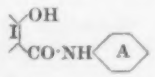
$$H{\cdot}NR^1{\cdot}(CH_2)_n{\cdot}X$$

(X = $-NR^2R^3$ or $-Y{\cdot}(CH_2)_n{\cdot}NR^2R^3$; Y = alkylamino, arylamino or piperazino; R^1, R^2 and R^3 = H or hydrocarbon; n = > 1) dyes more deeply with acid dyes than does the untreated nylon. Thus nylon is impregnated with 20% aqueous hexamethylenediamine at room temperature, wrung out and then heated at 218°C. for 1 hr. in an atmosphere of nitrogen. The treated pattern when dyed with C.I. Acid Green 25 absorbed three times as much dye as did an untreated pattern dyed in the same way. C.O.C.

Azoic Dyeings on Polyester Fibres

FH *BP* 809,221

Polyester fibres are dyed by applying simultaneously a diazotisable amine and a coupling component



(I = residue of naphthalene or carbazole; nucleus A may carry one or more Alk or OAlk groups, or when I = a carbazole residue, A may contain Hal) in presence of a carrier, e.g. tetrahydronaphthalene, followed by treatment in hot aq. nitrous acid. Thus, by applying 2-amino-4-nitroanisole and 3,2-hydroxynaphtho-*o*-toluidide at 90–100°C. from a bath containing tetrahydronaphthalene at a concentration of 4 cc./l., followed by treatment at 70°C. in a bath containing 4 g. of $NaNO_2$ and 5 cc. of 96% H_2SO_4 per litre, a yellowish-red is obtained. E.S.

Dyeing Polyolefin Fibres

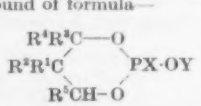
Vereinigte Glanzstoff-Fabriken *BP* 809,495

Excellent dyeings are obtained by use of a disperse or basic dye dissolved in an aromatic hydrocarbon, a hydrogenated aromatic hydrocarbon or an aromatic hydrocarbon substituted by one or more halogen atoms or methyl or ethyl groups. Thus polyethylene yarn was dyed for 1 hr. at 60°C. in a bath of *p*-xylene containing Celliton Fast Red Violet (C.I. Disperse Violet 1), dried and soaped. A deep violet-red dyeing was obtained. C.O.C.

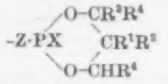
Phosphorinanyl Derivatives as Carriers in the Dyeing of Acrylic Polymers

Union Carbide Corpn. *USP* 2,859,086

High colour values are obtained on acrylic polymers with acid, including metal-complex dyes, disperse and vat dyes by either first treating the polymers with or carrying out dyeing in presence of 0·2% (on wt. of polymers) of a compound of formula—



(R^1, R^2, R^3, R^4 and R^5 = same or different, H or Alk; X = O or S; Y = Alk, Ar, alkaryl, halogenoalkyl, cyanoalkyl or



(Z = divalent radical having two terminal oxy groups)), e.g. 5-ethyl-2-(2-ethylhexyloxy)-2-oxo-4-propyl-1,3,2-dioxaphosphorinane. C.O.C.

Luminescence of Dyes in Viscous and Solid Solutions (IV p. 262)

Measurement of the Ratio of Lateral to Vertical Dye Diffusion Rates in Swollen Gelatin Coatings (IX p. 271)

IX— PRINTING

Chemical Foundations of Fast Colour Printing

F. R. Alsberg, W. Clarke, and A. S. Fern

J.S.D.C., **75**, 89–101 (Feb. 1959)

"Photon" Printing of Textiles

H. Schönefeld *Deut. Textiltech.*, **8**, 506–509 (1958): *Chem. Abs.*, **53**, 3701 (25 Feb. 1959)

Printing of textiles by illumination through negatives can be carried out by the following methods—(1) *With antidiazotates.* Diazotised primary aromatic amines exist in both syn and anti forms, both of which couple with naphthols, but only the syn form produces colour. The coupled antiform is changed to the coloured form by acids, heat or light. Exposed portions of the compounds have strong affinity for the fibre but the unexposed portions are readily washed off. (2) *With chromium salts.* When chromates are exposed to light in presence of colloids the final product is Cr_2O_3 and many colloids are rendered insoluble. When cloth is treated with chromates and such a colloid, then exposed to light through a negative and treated with mordant dyes, only the exposed portions are dyed. (3) *With chrome gelatin.* Here the unexposed emulsion is washed off with water at 38–40°C. and that remaining in the fibre is hardened by treatment with 40% formalin 50 g./l. (4) *Diazotype processes.* The goods are padded with diazotised 2-naphthylamine which changes to 2-naphthol when exposed to light. After exposure the goods are treated with a diazotised base. (5) *With silver salts.* The goods are treated with a paste containing $AgNO_3$ and FeC_2O_4. After drying in the dark, they are exposed to light through a negative, washed and developed with $Na_2S_2O_3$. (6) *With leuco dyes.* Basic dyes dissolved in HCl are reduced with Zn dust and precipitated with NaOH. The precipitate is shaken with ether. The goods are impregnated with this precipitate in presence of collodion and exposed to light through a negative. (7) *Cyano type processes.* Photon printing is also done with positives, when exposure would destroy some material present, e.g. a dyed ground. These methods include—(*a*) *With diazotised bases.* Exposure of a light sensitive diazotised base converts it to a phenol by the Sandmeyer reaction. This phenol will not react with azoic coupling components whereas the unexposed base does. (*b*) *Schoen's diazo type process.* The goods are impregnated with a 2% solution of diazotised 3-aminocarbazole or its $ZnCl_2$ salt, and exposed, either wet or dry, until the pattern develops as a bluish image on a yellow background. The unaffected diazonium compound is coupled with 1% 2-naphthol or any other azoic coupling component which does not have too strong a characteristic colour. (*c*) *Kogel's total photographic method.* The cloth is treated with the quinone diazides of *p*-aminophenol or *p*-aminodiphenylamine and dried. Light decomposes these compounds to colourless phenols. In the unexposed portions the diazides couple with the phenol formed in a subsequent brief exposure to form azo dyes. Complete development occurs in dilute caustic soda or in dry NH_3 gas. (*d*) *With oxidation dyes.* The cloth is padded with an ordinary dye which is destroyed by exposure to light. The exposed cloth is padded with aromatic bases in presence of mineral acids or by steaming. Radioactive isotopes can replace light as the source of energy. Instead of being used through a negative or positive, they can be used as a direct contact pattern on the impregnated cloth. C.O.C.

Spectrophotometric Investigation of Solutions of Dyes forming *H*-Aggregates

A. V. Borin and I. A. Pobedonostseva *Zhur. Nauch. i Priklad. Fot. i Kinematog.*, **3**, 256–266 (1958): *Chem. Abs.*, **53**, 931 (25 Jan. 1959)

Absorption spectra at 400–700 mμ. were measured of 5·5′-dimethoxy-3,3′-dialkyl-9-ethyl (I), 5,5′-dimethoxy-3,3′-dialkyl-9-methyl- (II), 5,5′,9-trimethyl-3,3′-dialkyl- (III), and 5,5′-dimethyl-3,3′-dialkyl-9-ethylthiacarbocyanine (IV) salts, two rhodacyanines (structural formulae given), a 3,3′-dimethylthiacarbocyanine (V) salt, and a 3,3′:8,10-tetramethylthiacarbocyanine (VI) salt, dissolved in water, CH_3OH and water containing 0·1–5·0% gelatin. Data are given in tables and graphs. The absorption maxima of the *H*-bands (hypsochromic bands caused by gelatin) of II, III, and V (4×10^{-5} M. in aqueous gelatin) are at 460, 460, and 470 mμ. respectively; the corresponding relative absorption coefficients are 2·50, 0·80, and about 0·8 respectively. No *H*-bands were found with I, IV, and V. The intensity of the *H*-bands rises to a maximum as the

gelatin concentration rises to a critical value which increases with the degree of aggregation of the gelatin. This is probably caused by greater adsorbance of micelles in the more highly dispersed gelatin. For gelatin concentrations above the critical value, the *H*-band intensity decreases with increase in gelatin in concentration, apparently because of accumulation of gelatin decomposition products in the solution. Substituent groups in the polymethin chain which disturb the planarity of the molecule, prevent *H*-aggregates of dyes forming. C.O.C.

Sensitisation of Mercurous Oxalate by Dyes
P. A. Van der Meulen and R. H. Brill
Phot. Sci. & Eng., **2**, 121–127 (1958): *Chem. Abs.*, **53**, 931 (25 Jan. 1959)

Adsorption isotherms of several sensitising dyes adsorbed on $Hg_2C_2O_4$ and the reflectance spectra of the adsorbed dyes are given. Adsorption is often accompanied by bathochromic shift of the maximum. Acriflavine (C.I. 46000), Acridine Orange (C.I. Basic Orange 14), Acridine Yellow (C.I. 46025), and Proflavine are good sensitisers and are strongly adsorbed; Pyronine B (C.I. 45010) and Acridine Red (C.I. 45000) are weak sensitisers and are weakly adsorbed. Order of sensitising in the phthalein dyes is: Erythrosine B (C.I. Acid Red 51) > Eosine Y (C.I. Acid Red 87) > Rose Bengal (C.I. Acid Red 94) > Phloxine B (C.I. Acid Red 92). Erythrosine B and Rose Bengal are more strongly adsorbed than Eosine Y. Several cyanine dyes are no better than dyes of the other classes. The spectral range of sensitisation of $Hg_2C_2O_4$ and Ag halide by a given dye is about the same. C.O.C.

Measurement of the Ratio of Lateral to Vertical Dye Diffusion Rates in Swollen Gelatin Coatings
R. B. Pontius and J. A. Wenrich
Phot. Sci. & Eng., **2**, 131–135 (1958): *Chem. Abs.*, **53**, 933 (25 Jan. 1959)

Lateral and vertical diffusion rates of Brilliant Alizarine Light Red B (C.I. Acid Red 58) in hardened gelatin coatings at pH 4·2 and 20°c. have been measured. Diffusion was stopped by rapid freezing and low temperature dehydration, dye penetration was measured in the dry sections with a specially-designed microdensitometer. No significant differences between diffusion rates in the lateral and vertical directions were found. C.O.C.

PATENTS

Binding Agent for Use in Dyeing and Printing Pastes
S *BP* 810,261

A binding agent for pastes containing pigments is obtained by incorporating a triglyceride of a fatty acid containing > 1 conjugated double bond in the molecule into an oil-in-water type emulsion of a polymeric or polymerisable compound containing at least one $CH_2{:}C<$ group and < 12 C and then completing the polymerisation or copolymerisation of any ingredients. Thus a 10% aqueous solution of polyvinyl alcohol (average mol.wt. 23,000, saponification index 100) (83 parts) is mixed in a colloid mill with China wood oil (6) and the condensate (1) of oleylalcohol (1 mol.)–ethylene oxide (24 mol.). To this mixture is added a 20% paste of C.I. Pigment Blue 15 (10). Prints produced with this product on cotton satin are dried at 60–70°c. and baked for 10 min. at 130°F. This yields brilliant prints of excellent fastness to washing, dry cleaning, and rubbing. C.O.C.

Fixing Images on Dye Cyanide Photosensitised Materials
L. Chalkley *USP* 2,855,300

The triphenylmethane dye images obtained by exposure of dye cyanide sensitised paper or cloth are given excellent fastness to light by treating them with a polyacid of tungsten or molybdenum, e.g. phosphotungstomolybdic acid, phosphotungstic acid, silicotungstic acid, phosphomolybdic acid, and silicomolybdic acid. C.O.C.

Activated Triphenylmethane Dye Cyanide of Good Stability to Ageing
L. Chalkley *USP* 2,855,303

Hydrophilic triphenylmethane dye cyanides of the phenolic and amino types form photosensitive combinations with hydrocolloids. These combinations have good resistance to ageing. This principle may be made use of in several ways, e.g. cotton cloth is impregnated with a 1% solution of the cyanide of Ethyl Green (C.I. 42590), squeezed between rubber rollers and dried in the dark. The dry cloth is then exposed behind a stencil to ultraviolet radiation of < 3350 A. to print a green design on a colourless ground. C.O.C.

Activated Aurin Cyanide
L. Chalkley *USP* 2,855,304

Aurin cyanide 4,4′,4″-trihydroxytriphenylacetonitrile, is colourless, difficultly soluble in water but readily soluble in dilute ammonia. It forms photosensitive combinations with hydrophilic colloids, which combinations have good resistance to ageing. Paper treated with a 2·5% solution of aurin cyanide (in 28% ammonia) and dried on exposure to radiation of < 3000 A. becomes orange. C.O.C.

Alginate Diammonium Phosphate Compositions as Thickeners for Printing Pastes
Kelco Co. *USP* 2,857,236

Addition to an alginate thickener of 10% diammonium phosphate on the anhydrous weight of the alginate, renders use of a hygroscopic sequestering agent unnecessary and yields a product which is stable during storage, and readily mixes with water. C.O.C.

Screen Printing
Gebr. Stork & Co's Apparatenfabriek *BP* 809,525

Improvement of the machine described in *BP* 718,459 (J.S.D.C., **71**, 108 (1955)) which makes possible choice of the length of the inoperative squeegee strokes so that they do not take place above the stencilled portion of the screen. By this means any printing paste that may drip off the squeegee cannot fall onto the stencilled portion of the screen. C.O.C.

A Contribution to the Theory of the Optical Sensitisation of Photographic Emulsions. Synthesis of *N*-Phenylbenzimidazoletrimethincyanines (IV p. 262)
Luminescence of Dyes in Viscous and Solid Solutions (IV p. 262)
Mechanism of Ink-splitting during Printing (V p. 266)
Flash Photolysis of Vat Dyes (VIII p. 269)
Durable Inlay Effects on Embossed Fabrics (X p. 272)

X—SIZING AND FINISHING

Textile Finishing—Problems of Today and Tomorrow
W. Gutmann J.S.D.C., **75**, 81–88 (Feb. 1959)

Waterproofing of Cotton Fabrics with Organosilicon Compounds
N. V. Kalugin and M. G. Voronkov
Zhur. priklad. khim., **31**, 1390–1397 (Sept. 1958)

Methyldiacetoxystearoxysilane $CH_3{\cdot}Si{\cdot}[O{\cdot}CO{\cdot}CH_3]_2{\cdot}O{\cdot}CO{\cdot}C_{17}H_{35}$, Na polymethylsiloxanolate, and polyethylhydroxysiloxane $[C_2H_5SiHO]_n$, can be used for imparting water-repellency to cotton fabrics. The methyldiacetoxystearoxysilane is applied from an organic solvent, such as CCl_4, the other two from aqueous solution and emulsion respectively. The cotton fabric, after impregnation with the waterproofing soln. or emulsion, is dried at a low temp. and then heated at 100–110°c. for 1–4 hr. or at 150°c. for a very much shorter time. The temp. and the time of "curing" can be decreased by use of small quantities of $Ti(O{\cdot}C_2H_5)_4$ as a catalyst. The employment of organosilicon compounds allows the waterproofing treatment to be combined with rotproofing, which can be done in the absence of Al salts, thus increasing the resistance of the fabric to light and weathering. T.Z.W.

PATENTS

Softening Man-made Fibres
Spinnfaser *BP* 810,040

Considerable saving in lubricant and improved behaviour of the fibres in subsequent processing is obtained if they are first treated with an oil-in-water emulsion containing 0·1–0·2 g./l. of a liquid lubricant of oil consistency, squeezed or centrifuged, and then the remainder of the lubricant is applied in a similar emulsion but of strength 70–200 g./l. C.O.C.

Water-repellent Finish
Bradford Dyers' Association *BP* 809,822

Addition to organosilicon compounds used for imparting water repellent finishes to textiles of an alkanolamine titanate, e.g. triethanolamine titanate, in the proportion

of 2·5–125·0% on the weight of the siloxane, greatly improves the water repellent finish they yield. C.O.C.

Water-repellent Finish
General Electric Co. *BP* 810,310

Organopolysiloxanes can be used as water repellent agents in absence of (1) Si-H-containing organopolysiloxanes, (2) catalyst, and (3) heat. Thus fabric is treated with an organopolysiloxane and then irradiated in a fast continuous process at room temperature, this irradiation requiring only a few seconds if an effective irradiation source is used. C.O.C.

Treating Textiles with Methyl Silicone Resin and *NN'*-Diphenylguanidine
R. E. Ellis *USP* 2,859,137

Use of a symmetrical *NN'*-di-hydrocarbon-substituted guanidine, e.g. diphenylguanidine, as catalyst enables silicone finishes or coatings to be cured at $> 100°$c. C.O.C.

Antiseptic Cotton Cloth
H. N. Vagenius *USP* 2,856,330

Cotton cloth is rendered resistant to development of unpleasant odours and/or rise in pH when exposed to perspiration, urine or other body secretions, by treating with an aqueous solution containing 0·05–1·0 milligram molecules (mol.wt. in mg.) of a copper salt for each 60 g. of cotton and 2–10 mg. mol. of disodium hydrogen phosphate per mg. mol. of copper salt. The pH of the solution is maintained at 3–6, time and temperature of treatment are not critical. The volume of the bath is 100–1000 c.c. or more. The treated material is colourless. Thus a 60 g. cotton napkin was wetted with 150–180 c.c. of water containing 0·2 g. soap, then immersed for 3–10 min. in 250 c.c. water containing 0·05 mg. mol. $CuSO_4$ and 0·1 mg. mol. of disodium pyrophosphate, the solution being brought to pH 5 with zinc silicofluoride. A treated and an untreated napkin were each impregnated with 90 c.c. urine and kept in a polyethylene bag for 6 days. The untreated napkin developed an unpleasant sour smell within 24 hr. followed shortly by rise in pH and generation of ammonia. The treated napkin did not smell or rise in pH before 4 or 5 days and generation of ammonia did not occur at all. C.O.C.

Cyanoethylation of Cotton
American Cyanamid Co. *USP* 2,857,239

Cotton cyanoethylated at 10–20°c. in presence of aqueous 8–12% caustic soda in a liquor of $> 14:1$ and < 1 part acrylonitrile to 4 parts of cellulose, yields a product showing increased tensile strength, improved resistance to degradation by heat and of better resistance to micro-organisms than the untreated cotton, but differs from cotton cyanoethylated in the usual manner in that it is not completely mildew proof. C.O.C.

Processing of Heat-shrinkable Textiles
Firestone Tire & Rubber Co. *BP* 809,916

The material passes first through a coating fluid and is then heated to 320–450°f. while being stretched to $> 21\%$ of its original length. Finally it is heated to 400–450°f. while being relaxed under control so that it loses 0–10% of its stretched length. This results in a product of improved dimensional stability in use. C.O.C.

Coated Fabrics
B.T.R. Industries *BP* 809,922

Some or all of the yarn used to form a fabric to be coated with a plasticised polymer is treated with a plasticiser for the polymer before the yarn is knitted or woven. This facilitates bonding of the coating to the fabric and the plasticiser also acts as a size for the yarn during wearing. C.O.C.

Durable Inlay Effects on Embossed Fabrics
Joseph Bancroft & Sons Co. *USP* 2,858,232

The fabric is impregnated with a thermosetting precondensate, dried to 8–12% moisture content and then passed through an embossing calender where simultaneously with the embossing the depressed portions of the cloth are coloured to produce a coloured inlay effect. The printing paste used to colour the inlay contains the precondensate of a modified alkyd resin. The embossed fabric is then baked to fix the resins. This results in clear and sharp inlay effects fast to washing and dry cleaning. C.O.C.

Metallising Synthetic-polymer Fibres
Deutsche Gold- und Silber Scheideanstalt vormals Roessler *BP* 809,958

The fibres are treated with metal vapour in a vacuum either before or after treatment with an agent which makes them resistant to swelling and/or water repellent. Thus excellent bonding of the metal is obtained on acrylic, polyamide or polyester fibres that have been treated with a silicone. C.O.C.

Drying Impregnated Porous Webs (I p. 259)
Durable Pleating of Wool Fabrics—Effect on Dyes (VIII p. 269)
Heat Fastness of Dyes on Synthetic Fibres (VIII p. 269)
Impregnated Fibre Webs (XI p. 273)

XI—PAPER AND OTHER CELLULOSIC PRODUCTS

Chemical Reactions of Fibrous Cellulose
G. E. Goheen *Tappi*, **41**, 737–742 (Dec. 1958)

Work done by the U.S. Dept. of Agriculture and others is reviewed, covering esterification, etherification, replacement of hydroxyl with halides, and oxidation with inorganic reagents (41 references). R.A.

Decomposition, Stability, and Re-formation of Ordered Structures of Cellulose during Re-precipitation
H. Dolmetsch *Das Papier*, **13**, 37–46 (Feb. 1959)

Characteristic types of structures, isolated from very dilute solutions of cellulose, are defined and compared with the corresponding elements of the unswollen fibre. Investigation of these regenerated structures back to the state of re-dissolution reveals rules for structural formation. The results may be used to explain the phenomena observed during the processing of concentrated cellulose solutions used commercially. R.A.

Yellowing of Pulp
H. Sihtola, B. Anthoni, Y. Hentola, I. Palenius, and N. E. Virkola *Paperi ja Puu*, **40**, 579–584 (Nov. 1958)

After reviewing the reasons for the yellowing of pulp, results are given of new investigations. Aldehydic carbonyls in the end units of the molecular chains do not affect the yellowing, nor does heating essentially change the copper number or the carboxyl content of cotton linters and periodate oxycellulose prepared from them. The carboxyl content of "dicarboxyl celluloses" obtained by chlorite oxidation from periodate oxycellulose is markedly reduced by heating. R.A.

Effect of Pigment Volume Concentration on Coating Properties
W. R. Willets and F. R. Marchetti *Tappi*, **41**, 743–749 (Dec. 1958)

A paper coating may be visualised as a spatial configuration of pigment particles of definite size, shape and distribution in a matrix composed of air and adhesive deposited on an irregular web of fibre. Irrespective of adhesive migration into the paper, the dried coating may be considered as consisting of distinct volumes of pigment, adhesive, and air or void. The size of the voids will vary with pigment particle size and distribution. The void volume influences the adhesive demand of a coating and, therefore, its pick resistance. Less void or pore volume should result in more efficient hold-out of gloss, inks, varnishes, and wax. Conversely, ink receptivity should be greater with greater pore volume. R.A.

Thermal Decomposition of Cellulose Nitrate studied by means of the Recording Thermal Balance
T. Abe, S. Tobisawa, and Y. Nomura *Bull. Chem. Soc. Japan*, **31**, 1041–1044 (Dec. 1958)

Darkening of Wood on Exposure to Light
A. W. Stout *Off. Dig. Fed. Paint Varn. Prod. Cl.*, **30**, 1423–1426 (Dec. 1958)

The darkening of wood by light is due to the photocatalytic degradation of its lignin content by radiation below 4000 A.; the nature of the reaction is not known. The treatment of panelled surfaces with light pigmented

finishes, or finishes incorporating ultraviolet absorbers, merely prolongs the time of darkening, and does not prevent it. J.W.D.

PATENTS

Paper Coating Composition
Corn Products Refining Co. *USP* 2,849,326

High solids contents, up to 70%, are achieved by the incorporation of polymers derived from starch. A composition having pH 8·0–9·5 is prepared from water (25–30), pigment (50–60) containing a little dispersing agent, and a partly hydrolysed, dried and, possibly, repolymerised starch of dextrin equivalent 5–50. The starch polymer should be ≮ 90% water-soluble. R.A.

Mineral-filled Papers
Rohm and Haas Co. *USP* 2,838,397

Fibre and filler retention in papermaking is improved by inclusion in the stock of 0·01–1% (by weight of dry fibre) of a quaternised high mol.wt., water-soluble polyester containing a basic group, e.g. a homopolymer of β-(*N*,*N*-dimethylamino)ethyl methacrylate quaternised by ethylene oxide. R.A.

Impregnated Fibre Webs
Wood Conversion Co. *USP* 2,843,505

Fibre webs which may be felts, or woven or non-woven, combed or carded structures and which are not self-supporting, are impregnated with a latex or similar while supported between two travelling screen carriers which are immersed in the impregnating bath and then passed through one or more sets of squeeze rolls. The effect of the dead spaces created by the screen structure of the carrier is counteracted if at least one pair of rolls is fitted with resilient surfaces capable of entering and filling the openings in the carrier screen at the height of the pressing step. R.A.

Ink-receptive, Oil-transparentised Paper
Standard Register Co. *USP* 2,851,378

Ordinary bond paper is rendered translucent or transparent, before or after typing, printing, writing, etc., by treatment with a soln. comprising 35–55% of an organic solvent, 10–25% castor oil, 20–50% of the monostearate ester of a polyethylene glycol and minor amounts of antioxidant and odour inhibitor. The product is useful as a master for duplicating processes. A recommended composition consists of 37·82% Carbowax monostearate, 18·91% cold-pressed castor oil, 43·22% denatured alcohol, 0·03% antioxidant, and 0·02% odour neutraliser. R.A.

Drying Impregnated Porous Webs (I p. 259)
Control of Slime-forming Organisms (III p. 260)

XII— LEATHER; FURS; OTHER PROTEIN MATERIALS

Action of Ethylene Sulphide on Collagen
A. Gill
J. Soc. Leather Trades Chem., **42**, 394–403 (Dec. 1958)

Ethylene sulphide polymerises within the structure of collagen and leather producing increased hydrothermal stability; this is attributed to the hydrophobic nature of the deposited polyethylene sulphide, and evidence suggests that cross-linkage with residues of the polypeptide chain is unlikely. J.W.D.

Rabbit Fibres. XX— Colouring and Crimp increase of Carrotted Fur
S. Ikeda and S. Okajima
J. Soc. Textile Cellulose Ind. Japan, **14**, 242–250 (April 1958)

Labelling of Proteins with Cellulose-reactive Dyes
R. Hess and A. G. E. Pearse
Nature, **183**, 260–261 (24 Jan. 1959)

The labelling of proteins with reactive dyes bearing a dichlorotriazinyl grouping is simple and is advantageous over other current methods. Reaction is carried out in 0·85% sodium chloride at pH 7·4 for 30–60 min. at 20–22°C. Unfixed dye was removed by dialysis against 0·06 M. phosphate-buffered saline of pH 7·4. Dye was used at a concentration of 1–10% of the protein. With serum albumin the labelled protein gave an electrophoretic pattern identical to that of the pure protein. With tissue sections good results are also obtained with some of the more highly coloured dyes, but best results are obtained with similar reactive compounds containing a fluorescent chromophore. Dyes containing the dichlorotriazinyl group (the type marketed by ICI) were found to be superior to dyes containing other reactive groups. W.J.M.

XIII— RUBBER; RESINS; PLASTICS

Carbon-chain Polymers and Copolymers. V—Copolymerisation of Acrylonitrile and Methyl Methacrylate in presence of Tributylboron
G. S. Kolesnikov and N. V. Klimentova
Izvestiya Akad. Nauk S.S.S.R., otdel. khim. nauk, (11), 1383–1387 (Nov. 1958)

Kinetics of the Polycondensation of Dicarboxylic Acid Chlorides with Dihydric Phenols
S. V. Vinogradova and V. V. Korshak
Doklady Akad. Nauk S.S.S.R., **123**, 849–852 (11 Dec. 1958)

Polyurethanes— Properties and Applications
J. M. Buist *Plastics Inst. Trans. J.*, **27**, 13–27 (Feb. 1959)

PATENT

Coating Polymeric Substances with Polymers
DuP *BP* 809,558

A polymeric film or other substrate is irradiated with high energy particle irradiation in a fluid atmosphere containing a compound capable of forming free radicals and is then immediately coated with an ethylenically unsaturated liquid monomer to effect polymerisation and bonding of the coating to the surface of the substrate. Thus polyvinyl fluoride film was irradiated under the beam of a Van de Graaf generator while immersed in aqueous 5% H_2O_2. The film and solution were subject to a particle irradiation dosage of 3,000,000 rep. The film was quickly removed, washed with water and immediately immersed for 30–60 sec. in a liquid styrene monomer. Finally it was dried for 30–90 min. at 25°c. The coated film had much improved printability. C.O.C.

Emulsions of Hardenable Resins (III p. 260)

XIV— ANALYSIS; TESTING; APPARATUS

Ion-exchangers in Detergent Analysis
P. Voogt *Rec. Trav. chim.*, **77**, 889–901 (Sept.–Oct. 1958)

Mixtures of the Na salts of alkylsulphuric or sulphonic acids, fatty acids, and non-ionic material are passed over a succession of three ion-exchangers ($[H^+]$, $[Ac^-]$, $[OH^-]$) and are thereby separated into weakly acid, strongly acid, and non-ionic fractions; these can be titrated potentiometrically and the contents and mol.wt. of the individual acids thus determined. Non-ionic constituents are determined gravimetrically. This is a method of approach rather than a general and universal procedure, and modification is essential in many cases, e.g. with cpd. that decompose during elution. A procedure is described for the analysis of the alcohol extract of a detergent and there is a diagram of the apparatus. J.W.D.

Chromatographic Qualitative Analysis of Oxidation Bases
N. V. Grigor'eva
Nauch.-Issledovatel. Trudy Nauch.-Issledovatel. Inst. Mekhovš Prom., **6**, 32–35 (1955); *Referat. Zhur., Khim.* (1956), Abstr. No. 56795; *Chem. Abs.*, **53**, 3700 (25 Feb. 1959)

Silica gel (0·1 mm. particles) washed, dried, and treated with $FeCl_3$ till it contained 0·02–0·04% Fe_2O_3, was washed with distilled water till free of chlorine ions and then dried at 110–120°c. A 0·5 mm. layer of the gel was placed in a plexiglas dish, 5 drops of the dye (the oxidised base) solution are dropped into the gel from a pipette, and after 2 min. 10 drops of distilled water are added. This yields a washed chromatogram which is characteristic for a particular dye and which is compared with standard chromatograms of known dyes. C.O.C.

Analysis of Solvent Dyes by Reversed-phase Paper Chromatography
B. R. Roy and A. R. Sundarajan
J. Proc. Inst. Chemists (India), **30**, Pt. 3, 157–160 (1958); *Chem. Abs.*, **53**, 3518 (25 Feb. 1959)

Filter paper impregnated with different proportions of medicinal paraffin was used as chromatogram and alcoholic solutions of different strengths as developer. C.O.C.

Isolation of Oil-soluble Coal-tar Colours from Foods
E. Mark and G. G. McKeown
J. Assocn. Offic. Agri. Chemists, **41**, 817–818 (1958): *Chem. Abs.*, **53**, 1573 (25 Jan. 1959)

Common oil-soluble azo dyes can be isolated from corn (maize) oil by solvent partition using *NN*-dimethylformamide and petroleum ether. The dyes are recovered unchanged in good yield and 99·8% of the oil is removed. C.O.C.

Analysing Rutile-Anatase Mixtures (C.I. Pigment White 6) by Measurement of their Dielectric Constants
F. Oehme *Farbe u. Lack*, **64**, 319–321 (1958): *Chem. Abs.*, **53**, 2928 (25 Feb. 1959)

Dielectric constants of powdered rutile and anatase were determined from capacitance measurements of these pigments immersed in acetone by using the extrapolation calculation of Kleinke [*Physik Z.*, **36**, 565 (1935)]. The value for rutile was 80 and for anatase 46. A Dekameter, type DK 03, was used with immersion cells, type MPI 4T. The range of the cells was extended with a shunting condenser. The calculations were based on the change in capacitance of the cell when powdered rutile–anatase immersed in acetone was used. To analyse mixtures, a calibration curve was made of the changes in capacitance when 5·6 g. samples of known mixtures of rutile and anatase were added to a cell filled with acetone. Mixtures of unknown composition could then be analysed in 3–5 min. with ± 2% accuracy. It was found that dry samples were necessary for accurate results to be obtained. C.O.C.

Modified Testing Procedures for Ultramarine (C.I. Pigment Blue 29)
W. F. Wyatt
J. Oil & Col. Chem. Assocn., **42**, 136–145 (Feb. 1959)

Time-saving methods are described for determining the particle-size distribution (by sedimentation) and the tinting strength (by reflectance measurement on a zinc oxide reduction in linseed oil) of Ultramarine pigments. Methods are also given for the measurement of acid resistance and water sensitivity of these pigments in paint films. For some pigments at least, the water absorption of a paint film containing a mixture of pigments of known water-absorption characteristics is in proportion to the quantities of each pigment present. J.W.D.

Pigment Optical Behaviour— Evaluation on a Physical Basis
P. B. Mitton and L. S. White
Off. Dig. Fed. Paint Varn. Prod. Cl., **30**, 1259–1276 (Nov. 1958)

The Kubelka–Munk scattering coefficient S is related to the refractive index, the particle size, shape, and distribution, and the degree of dispersion of the pigment. The Kubelka–Munk absorption coefficient K varies, for a given system, with wavelength. The tinting strength of white pigments is a function of S, whereas hiding power is a function of both S and K. The relative tone of white paints tinted grey is related to the variation of S with wavelength which, in turn, is related to the relative difference in particle size of the white pigments. The brightness of thick white films is a function of K/S with the green filter of a three-filter colorimeter and the tone is a function of K/S with the blue filter minus K/S with the red filter. A numerical example is given for two pigments one of which is bluer in tone in both a tinted paint and in thin white films over a black substrate, but is yellower in tone in thick white films. These phenomena follow directly from the S and K values of the pigments. J.W.D.

Opacity of Paint
W. R. Blevin *J. Oil & Col. Chem. Assocn.*, **41**, 850–861 (Dec. 1958); **42**, 94 (Jan. 1959)

The use of hiding-power boards to measure the opacity of paints is examined in the light of the Kubelka–Munk theory of light diffusion. Such results are shown to have little meaning unless the boards are suitably specified; a basis for establishing such specifications is given. J.W.D.

Gloss Scale for Paint Surfaces
M. C. Boshoff *J. Opt. Soc. Amer.*, **48**, 741–746 (Oct. 1958)

Just noticeable differences in gloss, as determined from the means of observer histograms, can be expressed in terms of differences in luminance values at specular reflection and are considered as gloss units in a gloss-specification system. R.B.B.

Longitudinal Sectioning of Fibres and Plastic Films
N. Iwanow and R. Schneider
Bull. Inst. Text. France, (77), 55–62 (Oct. 1958)

Short lengths of fibre, 0·2–0·3 mm. in length, are mounted in collodion on a stainless steel disc, the surface of which is accurately plane and parallel to the stroke of the microtome knife. Movement of the carriage is controlled by a small synchronous motor. Successive sections, 1–2 μ. thick, are cut parallel to the axis of the fibre. All the sections are usable. Films of Cellophane or plastic can be sectioned similarly. Photomicrographs of sectioned fibres, and photographs of the modified microtome, are given. J.C.F.

Chemical Analysis in the Textile Industry
J. M. Bather *Dyer*, **121**, 181–183 (30 Jan. 1959)

A review of applications, including two methods arising from unpublished work: these relate to the determination of soap in alkaline baths containing neutral salts, and to the determination of perborate in bleaching baths. J.W.D.

Blended Woollen Structures. IV— Analytical Methods of Blend Determination
M. J. Coplan *Text. Research J.*, **28**, 956–964 (Nov. 1958)

The use of two methods— densitometric and micro-analytical spectrophotometric evaluation— for the statistical evaluation of blend variation along the length of strands is described. S.B.D.

Preparation and Use of the Alkaline Iron-Tartaric acid-Sodium Complex for Viscometric Determination of the Degree of Polymerisation of Cellulose
K. Edelmann and E. Horn
Faserforsch. und Textiltech., **9**, 493–500 (Nov. 1958)

The preparation of the solvent is described. It is stable to air and light for several months. Solution of cellulose and rayon is complete in 30 min. Intrinsic viscosities in this solvent can be linearly related to weight-average D.P.s obtained by the nitrate method. W.R.M.

Quantitative Analysis of Mixtures containing Casein, Wool, and Viscose Rayon Staple Fibres
H. J. Kessler and H. Zahn
Textil Praxis, **13**, 1275–1278 (Dec. 1958)

A sample of material is hydrolysed with 30% H_2SO_4 at 105°c. The nitrogen content of the hydrolysate (estimated by the Kjeldahl method) is proportional to the amount of wool and casein in the mixture (P%). The proportion of cellulosic fibre is obtained by subtraction and corresponds to $(100-P)$%. The combined content of cystein and cystine in the hydrolysate, estimated by the method of Zahn and Traumann (*Melliand Textilber.*, **35**, 1069 (1954)), corresponds to the wool content in the mixture (W%). The nitrogen content of the mixture can now be apportioned between wool and casein fibres, and the amount of casein corresponds to $(P-W)$%. L.A.T.

Determining the Change in Dimensions of Wool Fabrics on Pressing
Bull. Inst. Text. France, (75), 89–92 (June 1958)

Tentative specification for a standard method. J.C.F.

Estimation of Fatty Matter in Wool Top
M. Van Overbèke, P. Outreman, and G. Mazingue
Bull. Inst. Text. France, (75), 27–42 (June 1958)

If the sample of top is previously dried (2 hr. at 105°c.), Soxhlet extraction with diethyl ether removes arachis oil, self-emulsifying mineral oil, and oleine practically quantitatively. Synthetic detergents which may be present are partially extracted, but the extract contains little soap. If the sample is at normal regain, free fatty acids are produced by hydrolysis of the soap, and the amount of material extracted by ether is significantly greater. Extraction with ethyl alcohol subsequent to extraction with diethyl ether removes soap, and the extraction is substantially complete after 4 hr. The pink coloration which sometimes develops in wool during alcoholic extraction is caused by impurities in the alcohol. It is not produced if pure alcohol is used. Of other compounds examined as solvents for the extraction of fatty matter (methylene dichloride, trichlorethylene, benzene, carbon tetrachloride, acetone), carbon tetrachloride gives results which agree best with those given by

diethyl ether. Methylene dichloride is the most efficient solvent for oxidised fatty material, and is markedly more efficient than diethyl ether. The solvents used in this work were of good commercial quality, and were not specially purified. J.C.F.

Alkaline Thioglycollate Extraction Test for Wool
F. G. Lennox *Text. Research J.*, **28**, 949 (Nov. 1958)

0·2-g. samples of wool are digested for 1 hr. at 50°c. in 20 ml. of 0·1 M. thioglycollate, prepared by diluting the acid with 0·5 M. potassium carbonate. Estimation of nitrogen in aliquots of the filtrate by a semimicro Kjeldahl procedure enables the percentage extraction of the wool to be determined. S.B.D.

Identification of Synthetic Fibres by an Embedding Process on the Kofler Hot-stage
R. Fischer and T. Kartnig
Z. ges. Textilind., **60**, (19), 830–832 (Oct. 1958)

Fibres are mounted in a non-volatile liquid of suitable refractive index on a Kofler hot-stage fitted to a polarising microscope. The temperature is raised until the refractive indices of fibre and mountant become equal: this temperature is reproducible and characteristic. Observations are made in monochromatic light both parallel and perpendicular to the plane of polarisation. Successive observations in a series of five liquids of increasing refractive index, together with m.p. determinations on certain fibres and a limited number of confirmatory tests, permit the identification of 36 commercial synthetic fibres. J.C.F.

Application of Spot Tests in the Examination of Synthetic Fibres—I
F. Feigl, V. Gentil, and E. Jungreis
Text. Research J., **28**, 892–894 (Oct. 1958)

Pyrolytic oxidation of synthetic fibres with manganese dioxide on the micro scale enables nitrogenous fibres to be detected by the Griess reaction test (diazotisation). If nitrogen is present, pyrolysis with sulphur enables the polyacrylonitriles to be characterised by the formation of thiocyanic acid and its detection by reaction with ferric salts, as polyamides do not give a positive test. If acrylamide is used as copolymer in acrylonitrile fibres instead of vinyl chloride it may be detected by absorbing Rhodamine B from cold aqueous or benzene solutions. S.B.D.

Bleached Worsted Tops—Chemical Investigation
K. Ziegler and H. Zahn
Melliand Textilber., **40**, 87–89 (Jan. 1959)

Wool samples untreated and padded with 0·5–1% H_2O_2 soln. and dried at 50°c. in < 1 min. are analysed for cystine and cysteine, cysteic acid, tyrosine, and tryptophan, pH of aqueous extracts, and solubility in alkali, acid, and urea–bisulphite. Slightly bleached wool can be differentiated from controls only by analysis for cysteic acid, this increasing by 17–44% of the original 0·19–0·25%. In normally bleached wool the increase was 174%, whilst the tryptophan content fell by 13%. S.M.J.

Colorimetric Properties of White Cotton Cloth
E. S. Shanley
Amer. Dyestuff Rep., **47**, P 445–P 449 (30 June 1958)

The AATCC Subcommittee on Bleaching has carried out work to try to obtain a numerical specification for the visual appearance of white linen cloth and of bleached cotton cloth. Three different spectrophotometers were used, and their results converted into C.I.E. co-ordinates for comparison. The results were given in terms of the reflectance (or *Y*-axis value), the dominant wavelength, and the % saturation, none of which gave consistent results within instruments nor agreement between instruments. The instruments were able to give relative rankings of white linen samples in agreement with visual rankings, but, as found by Hammendinger and Lambert, no simple correlation between lightness and visual ranking could be found for near whites. However, correlation could be detected between the saturation and the visual ranking. The value of the dominant wavelength was fairly constant at 575 mμ., the visual apparent reflectance being between 88 and 92% and the saturation between 2 and 4%. In further experiments on ageing samples, the yellowish shades showed that the colour stability of white linen is not very good. Unfortunately, differences in colour which could be detected by the human eye were not distinguishable on the instruments. The spectrophotometers were, however, useful for examining colour differences between samples. Tests carried out on typical "blued" samples showed a saturation of 0·5% or less, a dominant wavelength at 490 mμ., and a brightness of about 88%, which denote a sheeting very close to the neutral point in colour space. Human observers tended to assess the "blued" samples as "whiter" than ordinary bleached samples. In general, the stability of blued samples was fairly low, being subject to fading after two months even in the dark. L.P.

Colour-aptitude Test—Analysis of Scores
D. L. Tilleard
J. Oil & Col. Chem. Assocn., **41**, 797–806 (Nov. 1958)

An analysis of the scores recorded by 300 people subjected to the Colour-aptitude Test devised by the Committee of the Inter-Society Color Council (U.S.A.). It is concluded that, whilst the ability to discriminate small colour differences (revealed by this test) does not in itself indicate aptitude as a colour matcher in an industry, it is a very valuable attribute, and this test provides a useful method of sorting for potential colour matchers. Repetition of the test by the same person usually gives similar scores, though differently constituted. Experienced people do not score higher than average, and a test at the outset of a career will thus indicate likely performance at later stages. Women score slightly higher than men, probably because of the very low incidence of defective colour vision amongst women. Persons with defective colour vision score lower than colour-normal people, but they usually obtain excellent results on the blue and yellow rows. J.W.D.

Results obtained with the Colour-aptitude Test
J. M. Adams
J. Oil & Col. Chem. Assocn., **41**, 807–813 (Nov. 1958)

The Inter-Society Colour-aptitude Test was applied by the Printing, Packaging and Allied Trades' Research Assocn. (PATRA), to 25 women and 175 men, 11 of the latter being colour-defective. The grouping of the matches made around the correct match indicates that, in general, the test does measure how closely the candidate is making the matches required of him. It is doubtful whether the test gives any indication of inborn aptitude for colour-matching. The wide-ranging times taken by different people for the completion of the test bore no relation to the scores obtained, and appear to reflect only the degree of confidence of the matcher. J.W.D.

Quantitative Evaluation of Colour Perception—A Hypothesis
E. Hoyt De Kleine
J. Opt. Soc. Amer., **48**, 722–725 (Oct. 1958)

From the postulate that the physiological response, as distinct from the more usually considered psychophysical response, varies as the square root of the radiant flux received by the retina, the author, a physician, develops a transformation of C.I.E. values. Limited preliminary tests show that these physiological values correlate well with observer colour response, as represented by the Munsell system. R.B.B.

Accuracy of Tristimulus Computations
G. W. Ingle *J. Opt. Soc. Amer.*, **48**, 761 (Oct. 1958)

Short letter emphasising the limitations of commonly used methods of tristimulus integration, especially regarding wavelength intervals employed, and pointing out the necessity for checking automatic integrators for the most highly selective transmittance curves, which concentrate transmittance at those wavelengths at which the tristimulus functions exhibit minimal values. R.B.B.

Quantitative Relation between Chromaticity Differences and Luminance Differences
E. M. Lowry and J. J. DePalma
J. Opt. Soc. Amer., **48**, 820–827 (Nov. 1958)

Differences between colours due to stimulus differences of chromaticity, of luminance, or of both may be reduced, or even eliminated, when viewed simultaneously in the presence of a veiling luminance. This destruction of contrast by glare suggests a means of correlating the two parameters responsible for colour differences. Preliminary work established this hypothesis. R.B.B.

Incomplete Integrating Sphere
O. E. Miller and A. J. Sant
J. Opt. Soc. Amer., **48**, 828–831 (Nov. 1958)
Development of a theory of the integrating sphere and its experimental investigation. R.B.B.

Colour Projection System
W. E. Glenn *J. Opt. Soc. Amer.*, **48**, 841–843 (Nov. 1958)

Cube-root Colour-coordinate System
L. G. Glasser, A. H. McKinney, C. D. Reilly, and P. D. Schnelle *J. Opt. Soc. Amer.*, **48**, 736–740 (Oct. 1958)
Simplification of the Adams chromatic-value system and of its modified version (Saunderson and Milner) is acheived empirically by using a cube-root value function. The modified system allows much simpler calculations of colour differences and a simple slide rule is described for this purpose. Correlation with the Munsell system and implications are discussed. R.B.B.

Colour Control in Dyed Union Fabrics
K. Murata and T. Ogihara
J. Soc. Textile Cellulose Ind. Japan, **13**, 693–702 (Oct. 1957)
Computations in N.B.S. units of colour differences from C.I.E. specifications to control the dyeing properties of viscose–acetate rayon union fabrics require so much time that the following simple inspection method should be considered. Colour differences are obtained from the reflectances at a given wavelength on the spectral reflectance curve. Blue dyes in general use were chosen for the inspection method because they have smaller MacAdam ellipses. Three identical samples of a union fabric were separately dyed with the direct dye Azine Brilliant Blue 6B conc. (C.I. Direct Blue 1) or the disperse dye Diacelliton Fast Brilliant Blue BF, and then the reflectance was determined at 480, 580, and 650 mμ. in turn—

For the undyed fabric $\gamma_t = 0{\cdot}56\left(1 - \frac{R_{480}}{R_{580}}\right) + 1{\cdot}11$
For the direct dyed fabric $\gamma_t = 0{\cdot}92R_{580} + 1{\cdot}3$
For the disperse dyed fabric $\gamma_t = 0{\cdot}77R_{650} + 1{\cdot}0$

where γ_t is the colour difference in MacAdam ellipse units and R is the reflectance at the subscript wavelengths. (*16 Tables and 7 Fig. in English.*) L.P.

Relation between Dye adsorbed and Reflectance of Dyed Viscose Rayon
K. Murata and T. Ogihara
J. Soc. Textile Cellulose Ind. Japan, **13**, 703–707 (Oct. 1957)
There is a correlation between the amount of dye adsorbed by viscose rayon and the reflectance, which also coincides with visual observation. A simple method of determination by reflectance on the dyed fibre at 580 mμ. is suggested. L.P.

Some Visual Functions of a Unilaterally Colour-blind Person
E. Berger, C. H. Graham, and Y. Hsia
I— Critical Fusion Frequency in Various Spectral Regions *J. Opt. Soc. Amer.*, **48**, 614–622 (Sept. 1958)
II— Binocular Brightness Matches in Various Spectral Regions
Ibid., 622–627

Relative Spectral Energy Distribution of Standard Light Sources for Colorimetry
B. Hisdal *J. Opt. Soc. Amer.*, **48**, 608–613 (Sept. 1958)
Tabulated data in common use for the spectral energy distributions of C.I.E. source A are those of a black-body radiator at 2854°K. Tungsten-filament lamps used as practical light sources produce different relative distributions, depending on filament geometry, owing to the selective spectral emissivity of the metal. The effect of this has been computed for a ribbon-filament lamp. It is unlikely to introduce chromaticity errors of the order of just noticeable visual steps, except possibly in work with a large field of view. It may be possible to find a type of coiled filament with a relative energy distribution considerably closer to the Planckian curve than that found for the ribbon-filament type. Alternatively, tabulated data for source A could be made to refer to the ribbon-filament lamp, rather than to a black-body radiator; i.e. source A could be redefined. Similar conclusions apply to the associated sources B and C. R.B.B.

Comparison of Munsell and MacAdam Colour Spaces
H. R. Davidson and H. Hemmendinger
J. Opt. Soc. Amer., **48**, 606–608 (Sept. 1958)
A contribution to the work of the O.S.A. Uniform Colour Scales Committee. Calculations based on "MacAdam space" usually agree better with visual estimates than do calculations based on Munsell renotation space, and the two are far from identical. Final assessment of the accuracy of the two systems is not yet possible. R.B.B.

Davidson Colour-matching Method
F. Bryant *Text. Research J.*, **28**, 811–812 (Sept. 1958)
The application of the formula—

$$F_M = \frac{a}{A}F_1 + \frac{b}{B}F_2 + \frac{c}{C}F_3 + \ldots + F_0\left(1 - \frac{a}{A} - \frac{b}{B} - \frac{c}{C} - \ldots\right)$$

to the matching of ink draw-downs is described. $F = (1 - R)^2/2R$, R being the reflectance of the surface at a given λ, and subscripts M, 1, 2, 3 . . . refer to a mixture and its components respectively. F_0 is a correction factor for the uncoloured material and can normally be neglected. A, B, C . . . denote the concn. for which the spectrophotometric curves of components 1, 2, 3 . . . were obtained, and a, b, c . . . denote the concn. of components 1, 2, 3 . . . in the mixture M. The method can be applied to the matching of dyeings. S.B.D.

Spacing of the Blue Light-fastness Standards
L. F. C. Friele J.S.D.C., **75**, 44–45 (Jan. 1959)

Light-fastness Testing using the Xenon Arc
H. Toepffer
Melliand Textilber., **39**, 1246–1251 (Nov. 1958)
Xenon Lamp XE 1500, Xenotest W, and the xenon arc spectrum are illustrated. Light-fastness testing is critically reviewed. Glass used with daylight exposure cuts off ultraviolet radiation, giving too high a rating for sensitive dyes, e.g. those containing nitro groups. Boosted tungsten-filament lamps, suitably filtered, simulate daylight, but are too slow, requiring 6 months for standard 7 to fade. The xenon arc spectrum, after filtering out radiation of $\lambda < 295$ mμ., is nearer to daylight than other practicable light sources; its ultraviolet intensity (2% of the total) approximates to the maximum possible with daylight. Assessments with the xenon arc therefore correspond to exposure in daylight of maximum ultraviolet content, and will give lower ratings than daylight exposures under glass; ratings comparable with the latter are given when ultraviolet-absorbing filters are employed with the instrument. Heat arising from the infrared radiation of xenon lamps, greater than that of daylight, is dissipated by cooling water and alternating light and dark periods. The latter permit recovery of the patterns, as during night in Nature. Voltage variations affect intensity, but not spectral distribution of xenon (unlike carbon) arc radiation, so that evaluation according to hours of illumination would be more consistent with the former. Exposure with the blue standards is preferred. Spectral distribution is liable to change after 1500 hr. use. Hours required for the first perceptible change in the blue standards are— (1) 1–1·5, (2) 2–2·5, (3) 3–3·5, (4) 4–5, (5) 8–10, (6) 20–30, (7) 60–70, (8) 160–180. R.H. of surface of specimens is 20–25% unless they are very strongly infrared-absorbing. In Xenotest W higher humidities can be produced, and weathering tests can be carried out. S.M.J.

Chromatographic Analysis of Fractions from Primary Flax Cells (VI p. 267)
Fast Solid Dyeings on Cotton (VIII p. 268)
Heat Fastness of Dyes on Synthetic Fibres (VIII p. 269)

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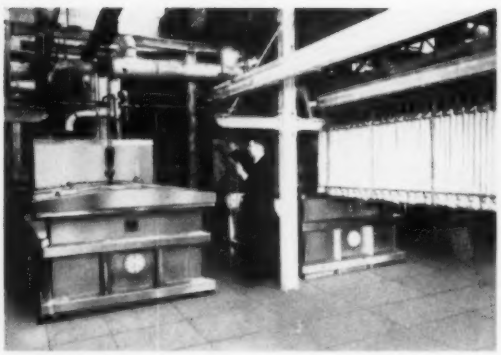

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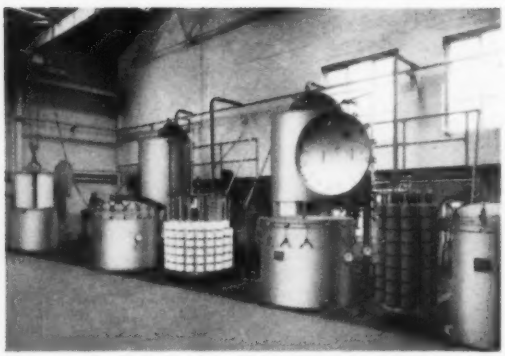

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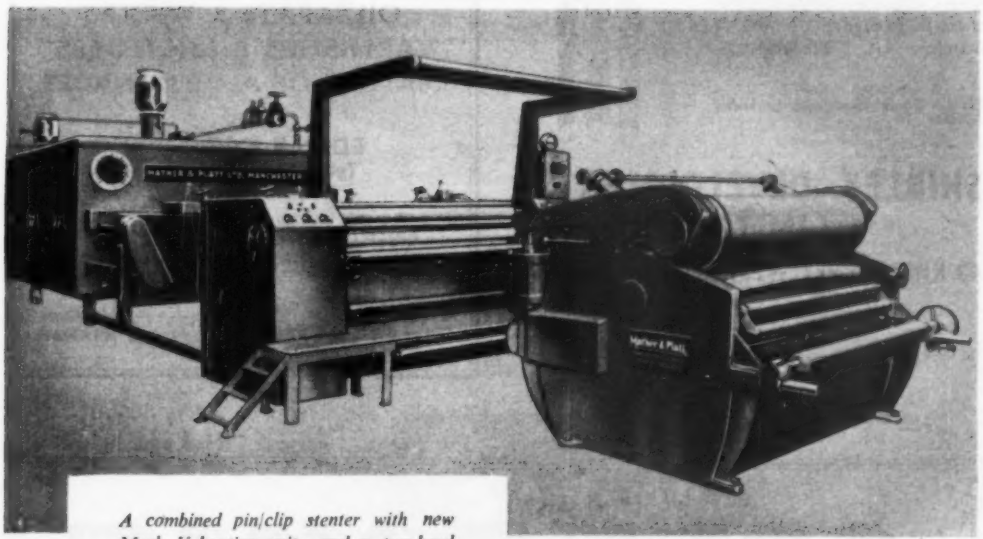

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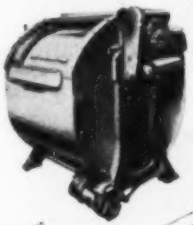

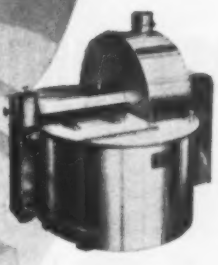

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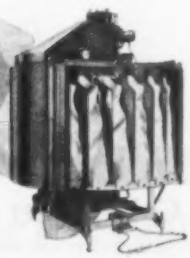

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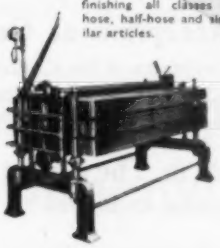

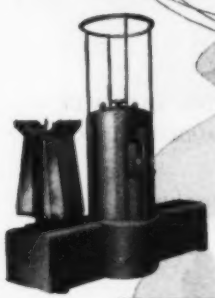

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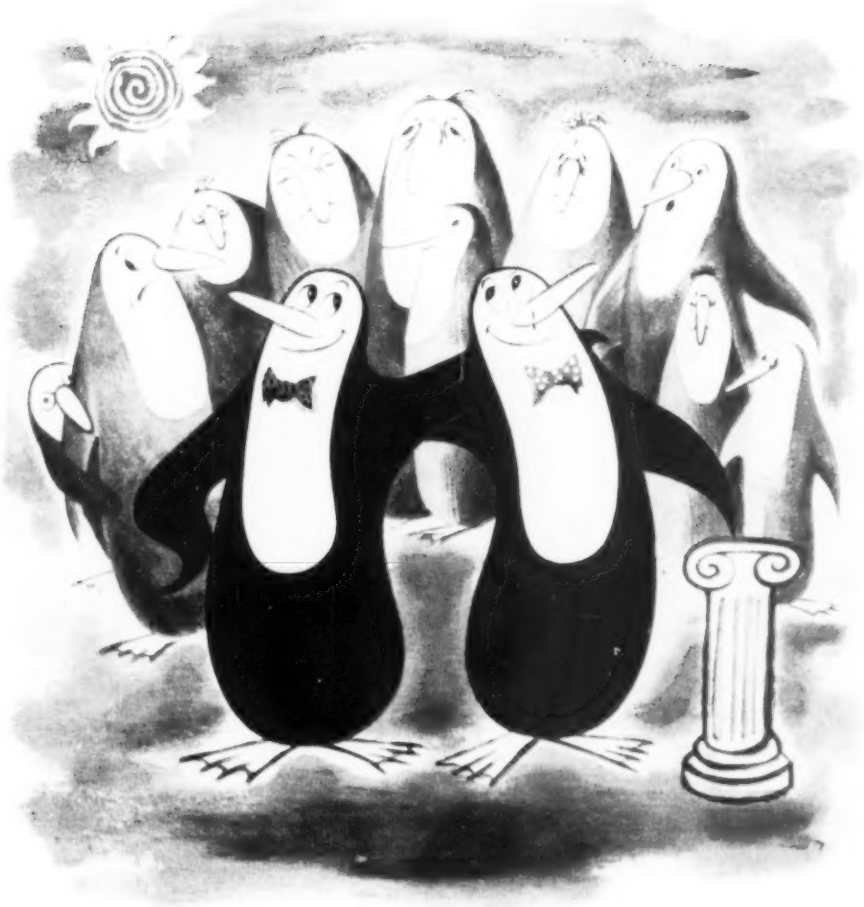

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